

Ed McKinnon  
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01852

December 6, 1999

Dear Mr. McKinnon:

**Re Correction on Proj# 26046, PG&E (PASC Submission # 9H0535):**

Regretfully, we must inform you that there were calculation errors on some of the draft ASTM mercury speciation data reported to you in the past. The portions that were impacted are the KCl and KMnO<sub>4</sub> impinger data which were reported high by a factor of 9/4<sup>th</sup> relative to the correct value.

The error occurred where a correction factor for sample vs standard prep of 1.5 was applied to the data in the wrong direction - a multiplication rather than a division. The 1.5 factor compensates for a 1.5-fold dilution of the standards during the preparation of these digested standards.

Enclosed is a revised report that presents the final results to the correct values. In regard to the corresponding data packages; the concentrations on the corresponding run sequence tables are incorrect since the dilution factors were entered as 1.5 (or appropriate dilutions of this value) rather than 0.667. The corresponding final ug of mercury per train portion as reported on the run sequence tables and on the validation tables have been reported high by the factor of 9/4<sup>th</sup>. I have enclosed copies of example pages from the original data package in which these errors are present. Instead of correcting each erroneous page of the original package, I request that with the data package as submitted to you prior, you include this letter of explanation and the enclosed revised report.

Of course quality data is of great concern to Philip and since this error has past several review stages before it has been identified, our review processes must be re-evaluated. We will also keep you informed as to the appropriated corrective actions that we implement to ensure that such an error will not repeat.

Should you have any questions in this regard please do not hesitate to contact me at extension 236 or the QA/QC scientist, Gerry Bengert, at extension 248.

Yours truly,



Ronald A. McLeod, Ph.D., C.Chem.  
Principal Scientist

cc: Gerry Bengert



January 21, 2000

U.S. Environmental Protection Agency  
Emission Measurement Center  
Interstate 40 and Page Road  
4930 Old Page Road  
Room Number E-108  
Durham, NC 27709

Attention: Mr. William Grimley  
Electric Utility Steam Generating Unit Mercury Test Program

Subject: Mercury Emissions Test Program of the Pulverized Coal Fired Boiler  
at the Logan Generating Station, Swedesboro, New Jersey  
(TRC Reference No. 26046)

Dear Mr. Grimley,

On behalf of PG&E Generating Company, TRC Environmental Corporation is submitting three (3) copies of a final report entitled, *"Mercury Emissions Test Program of the Pulverized Coal Fired Boiler at the Logan Generating Station, Swedesboro, New Jersey, Revision 1"*, dated January 2000.

The original report was submitted on November 24, 1999. TRC was notified, by Philip Analytical Services Corporation (PASC), on December 6, 1999 of an error in the laboratory results reported for KCL and KMnO<sub>4</sub> impingers. As a result, the original report should be considered invalid and not utilized for evaluation of the Logan test program. Copies of the original report should be discarded to avoid future confusion.

Emission results presented in the current document have been calculated based upon revised data packages submitted by PASC. A copy of the letter from PASC explaining the error that was made is provided as an attachment.

Should you have any questions regarding this change, please do not hesitate to call me at (978) 656-3550.

Sincerely,  
TRC Environmental Corporation

Michael P. Martin  
Project Manager

enclosures

cc: Mr. A. Rayner Kenison, PG&E Generating Company  
Mr. Thomas Fromm, PG&E Generating Company  
Ms. Valarie Gill, PG&E Generating Company

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TRC Project No. 26046-0040

January 2000

## **FINAL REPORT**

**Mercury Emissions Test Program of the Pulverized Coal Fired Boiler  
at the Logan Generating Station  
Swedesboro, New Jersey  
Revision 1**

Prepared for

PG&E Generating Company  
Logan Generating Station  
U.S. Route 130 South, P.O. Box 169C  
Swedesboro, New Jersey 08085-9300

Prepared by

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Boott Mills South  
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### **DISCLAIMER**

This report is intended for use solely by PG&E Generating Company for the specific purposes described in the contractual documents between TRC Environmental Corporation and PG&E Generating Company. All professional services performed and reports generated by TRC have been prepared for PG&E Generating Company's purposes as described in the contract. The information, statements and conclusions contained in the report have been prepared in accordance with the work statement and contract terms and conditions. The report may be subject to differing interpretations and/or may be misinterpreted by third persons or entities who were not involved in the investigative or consultation process. TRC Environmental Corporation therefore expressly disclaims any liability to persons other than PG&E Generating Company who may use or rely upon this report in any way or for any purpose.

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## **SECTION 1.0**

### **INTRODUCTION**

#### **1.1 SUMMARY OF TEST PROGRAM**

##### **1.1.1 Problem Definition and Background**

The United States Environmental Protection Agency (USEPA), in its "Study of Hazardous air pollutant emissions from Electric Utility Steam Generating Units--Final Report to Congress", stated that mercury is the Hazardous Air Pollutant (HAP) of greatest potential concern from coal-fired utility steam generators and that additional research and monitoring are merited. The USEPA also listed a number of research needs related to mercury emissions (e.g., how much is emitted from various types of units; how much is divalent vs. elemental mercury; and how do factors such as control device, fuel type, and plant configuration affect emissions and speciation). After reviewing the report, the Administrator concluded that obtaining additional information was appropriate and necessary for subsequent regulatory decisions. Specifically, the data will provide the USEPA with updated information on the total amount of mercury emitted from electric utility steam generating units and on the speciation and controllability of such mercury.

The USEPA, under its authority of Section 114 of the Clean Air Act (CAA), is requiring all coal-fired electric utility steam generating units to provide certain information under an Information Collection Request (ICR) that will allow the Agency to calculate the annual mercury emissions from each such unit and subsequently determine whether it is appropriate and necessary to regulate the mercury emissions from electric utility steam generating units. Section 112(n)(1)(A) of the CAA allows the Administrator to regulate the electric utility steam generating units if it is found that such regulation is appropriate and necessary after the results of the ICR are reviewed.

The ICR was approved on November 13, 1998 by the Office of Management and Budget and consists of three parts. In Part I, all units were required to submit background information on the coal fired, and unit descriptions, including operations and control devices. In Part II, all units



were required to sample the coal fired over each month at least three times, for the 1999 calendar year and analyze the samples for mercury, chlorine, gross heating value and proximate analysis. For participation in Part III, speciated mercury emission testing, the agency statistically selected units based on coal type, control device, and operations. Emissions testing was conducted utilizing the most current revision of the DRAFT Ontario Hydro Mercury Sampling Method. This method is a modification of USEPA Method 29 in 40 CFR 60 Appendix A.

The units selected for Part III were notified in writing by the USEPA. Units not selected were eligible to participate in Part III on a voluntary basis. PG&E Generating Company (PG&E Gen) elected to participate in Part III on a voluntary basis at the Logan Generating Station. The purpose of PG&E Gen's voluntary participation was two fold:

- to contribute to improved accuracy and completeness of the EPA's mercury ICR results; and
- to develop specific emissions data for several of PG&E Gen's coal fired electric utility steam generating units.

TRC Environmental Corporation (TRC) of Lowell, Massachusetts was retained by PG&E Gen to conduct the mercury emissions test program on the Pulverized Coal (PC) Boiler at the Logan Generating Station.

#### **1.1.2 Facility Information**

The facility is located in Swedesboro, New Jersey and is designated Air Pollution Control Plant ID No. 55357 by New Jersey Department of Environmental Protection (NJDEP). The stack ID No. is NJ001 and the permitting number is 01-94-2523.

Sulfur Dioxide (SO<sub>2</sub>) and Particulate Matter (PM) emissions from the unit are controlled by a spray dryer absorber (SDA) and a fabric filter bag house. Nitrogen Oxide (NO<sub>x</sub>) emissions from the unit are controlled through a Selective Catalytic Reduction (SCR) system. Mercury emissions testing was conducted in the inlet to the SDA, downstream of the SCR, and in the baghouse

exhaust stack to determine speciated mercury emissions prior to and following the facility emission controls.

## **1.2 PROJECT ORGANIZATION**

### **1.2.1 Purpose/Background**

The purpose of the project organization was to provide a clear understanding of the role that each party would play in the study and to provide lines of authority and reporting.

### **1.2.2 Roles and Responsibilities**

Figure 1-1 presents PG&E Gen's organization chart for this program showing the individuals responsible for each element of the overall program and the primary lines of communication.

#### **1.2.2.1 *PG&E Generating Company***

Mr. A. Rayner Kenison was the PG&E Gen Program Coordinator. He provided the overall program coordination amongst the Plant Program Coordinator, the USEPA Emissions Measurement Center, and TRC Environmental Corporation. The PG&E Gen Program Coordinator reviewed the Quality Assurance Program Plan (QAPP), the Site Specific Test Plan (SSTP), each test report, and submitted the final versions to the USEPA Emissions Measurement Center.

Mr. Thomas Fromm, Environmental Manager for the Logan Generating Station, served as the Plant Program Coordinator and directed the test program for the facility. Mr. Fromm acted as the primary contact with EPA/TRC and designated the appropriate PG&E Gen personnel to coordinate plant operations with the emission test program. The PG&E Gen Plant Program Coordinator coordinated the unit operations, the coal sampling, and emissions tests during each test run.

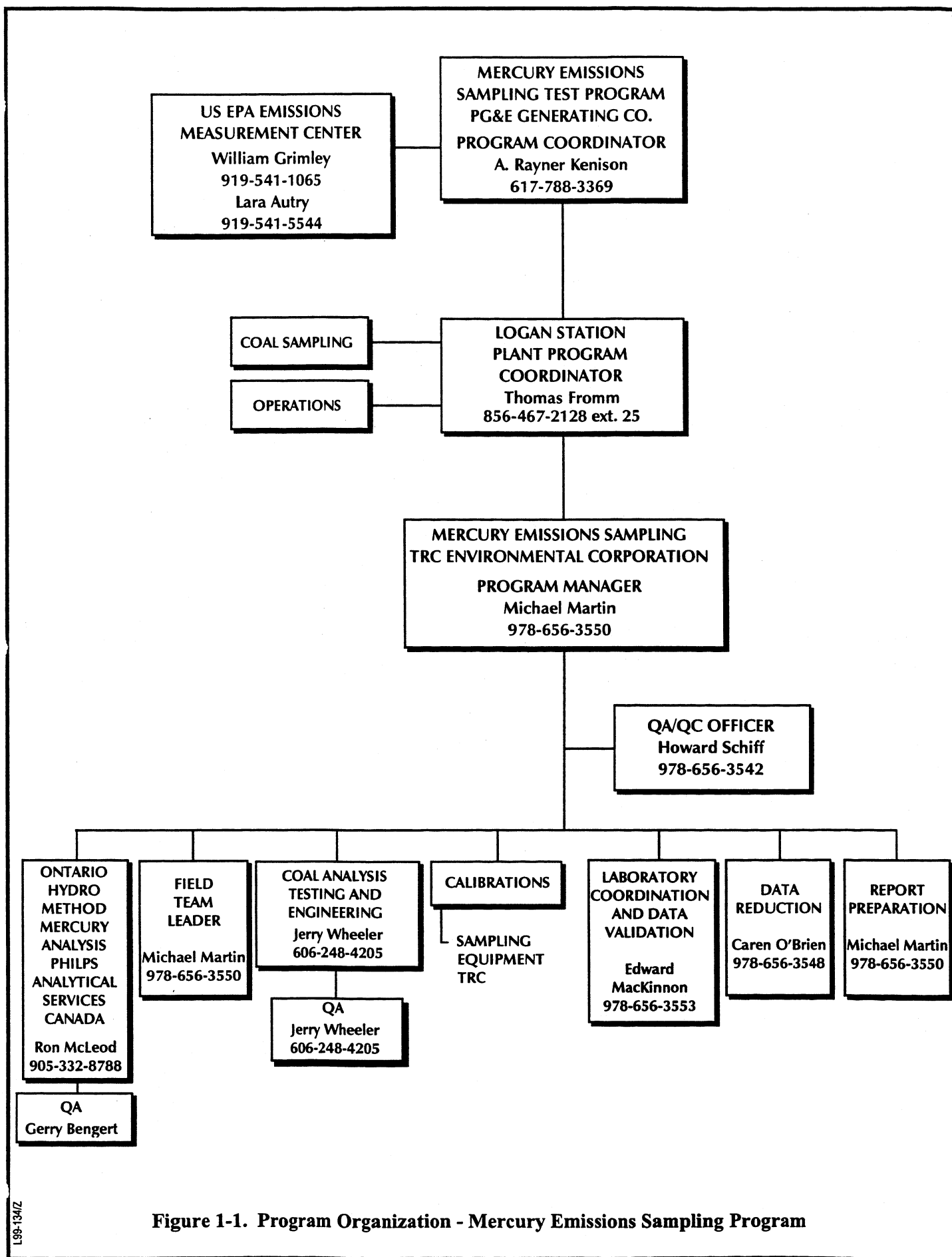


Figure 1-1. Program Organization - Mercury Emissions Sampling Program

#### *1.2.2.2 TRC Environmental Corporation*

TRC Environmental Corporation conducted the mercury emissions testing program for PG&E Gen.

##### *1.2.2.2.1 TRC's Program Manager*

Mr. Michael Martin, of TRC's Air Measurements Department, served as TRC's program manager and the primary point of contact with PG&E Gen for this project. TRC's field crew operated under the direct supervision of Mr. Martin. Mr. Martin coordinated the field crew's activities with the designated PG&E Gen personnel to complete the program on schedule and in accordance with the EPA approved Site Specific Test Plan (SSTP).

The Program Manager had the full responsibility and authority from both a technical and administrative standpoint for the successful conduct of this work. He was the principal point of contact with the PG&E Gen Program Coordinator for all matters relating to contract performance and technical progress.

Working with TRC's Laboratory Coordinator, TRC's Program Manager managed the assignment of analytical work to the analytical laboratories. Ultimately, TRC's Program Manager was responsible for assuring that all tasks were completed on schedule and within budget, while maintaining the quality objectives of the program. To do so, TRC's Program Manager carried out the following functions:

- Administered program activities within the TRC team (s).
- Coordinated activities within the TRC team(s).
- Attended program meetings.
- Conducted pretest site specific surveys.
- Effectuated corrective actions which included quality, budget and schedule maintenance measures.

- Interacted with the sampling teams to ensure proper performance of the test procedures.
- Communicated directly with the PG&E Gen Program Manager.
- Prepared or reviewed Site Specific Test Plans
- Reviewed the QAPP.
- Prepared or reviewed the site specific test reports.
- Reviewed the data validation and reduction.

In summary, TRC's Program Manager ensured that the program was effectively staffed, managed, coordinated and satisfactorily completed.

#### *1.2.2.2.2 TRC's QA Officer*

Program Quality Assurance / Quality Control was under the direction of Mr. Howard F. Schiff, Program QA Officer. He was responsible for ensuring that all program deliverables adhered to the highest quality principles. He reported programmatically to the Program Manager, but he derived his authority from the TRC Air Measurements Manager.

TRC's QA Officer initiated or followed up on corrective actions and aided in the preparation of the section of the site specific final report summarizing QA/QC activities, problems identified and corrective actions taken.

TRC's QA Officer carried out the following functions:

- Implemented all QA procedures.
- Prepared or reviewed the QAPP.
- Reviewed and approved each Site Specific Test Plan (SSTP) prior to submittal.
- Ensured that all required equipment calibrations were conducted prior and subsequent to each field test.
- Provided written summaries of Program QC activities for submission to the Program Manager.

- Advised technical staff of appropriate QC measures and corrective actions, prepared QC procedure write-up, as needed.
- Assisted in data analysis.
- Reviewed Site Specific Final Test Reports.

#### *1.2.2.2.3 TRC's Laboratory Coordinator*

Laboratory coordination and data validation were under the direction of Mr. Edward MacKinnon, who carried out the following functions:

- Acted as the laboratory coordinator between the sampling team(s) and the analytical laboratories.
- Communicated the specific analytical QC requirements to the laboratories.
- Supervised the schedule and budget for the laboratories.
- Received, validated, and distributed the laboratory data.
- Assisted in data analysis.
- Assisted in report preparation.

#### *1.2.2.2.4 Field Team Leader's Responsibilities*

The Field Team Leader coordinated the activities of the sampling team. The Field Team Leader was responsible for the following functions:

- Supervised the source sampling train operators.
- Coordinated the sampling program with the Plant Program Coordinators.
- Assisted the train operators in trouble-shooting and maintaining the sampling trains.
- Collected all sampling train data sheets, determined isokinetic ratios, determined acceptability of train leak checks and ensured that each train was operated in accordance with the EPA sampling protocol.

- Oversaw the recovery, packing and shipping of the samples to the respective analytical laboratory.
- Informed the TRC and PG&E Gen Program and Plant Program Coordinators on which sampling runs met all validating criteria and if not, determined if additional sampling runs were to be conducted.

#### **1.2.2.3      *Analytical Laboratory***

The contracted analytical laboratories (Phillips Analytical Services (Phillips) and Commercial Testing and Engineering) were responsible for sample analysis and assisting with data reporting. The contracted laboratories were responsible for conducting the analyses in accordance with the methods and procedures specified in the SSTP and the QAPP. Specifically, Phillips analyzed the Ontario Hydro Mercury train samples and Commercial Testing and Engineering analyzed the as-fired coal samples and the flue gas desulfurization media samples.

The Laboratory Managers were responsible to ensure that the QAPP was followed. In summary, the Laboratory Managers performed the following duties:

- Ensured that laboratory services were available to support the sample analysis.
- Ensured that the Program Quality Assurance Program Plan was followed.
- Ensured that the laboratory QA/QC procedures were implemented.

## SECTION 2.0

### FACILITY AND SAMPLING LOCATION DESCRIPTIONS

#### 2.1 PROCESS DESCRIPTION AND OPERATION

The Logan Generating Station is a 230 MW pulverized coal fired cogeneration facility which exports 50,000 lbs/hr of steam to a host facility. The plant is equipped with a flue gas cleaning system (FGCS) comprised of two 50% capacity lime injection spray dryer absorbers followed by a fabric filter (baghouse). The spray dryer absorbers and baghouse serve to reduce SO<sub>2</sub> and particulate emissions from the PC boiler. Low NO<sub>x</sub> burners with over-fire air and selective catalytic reduction (SCR) are used for NO<sub>x</sub> emission reduction. Oil ignitors are used for boiler warmup, coal burner ignition, and coal flame stabilization. An air flow schematic of the process is presented in Figure 2-1.

The facility is equipped with a continuous emission monitoring system (CEMS). The CEMS measures and reports opacity, O<sub>2</sub>, CO<sub>2</sub>, SO<sub>2</sub>, NO<sub>x</sub>, CO, and ammonia levels in the exhaust gas. Sulfur dioxide emissions are measured at both the FGCS inlet and outlet to provide data for FGCS control and to calculate SO<sub>2</sub> removal efficiency of the system. The CEMS data are also used to control the injection rate of ammonia.

The auxiliary boiler exhaust enters the main stack prior to the PC boiler test port location. As a result, the auxiliary boiler did not operate during the PC boiler testing. The 68,000 lbs/hr (77 MMBtu/hr) oil fired auxiliary boiler is normally used for process steam when the PC boiler is unavailable.

##### 2.1.1 Operating Schedule

During the test program, the PC boiler was operating at a "steady-state" load condition throughout each test run. The steady-state load represented the maximum capacity (+/- 5%) of the source being tested.



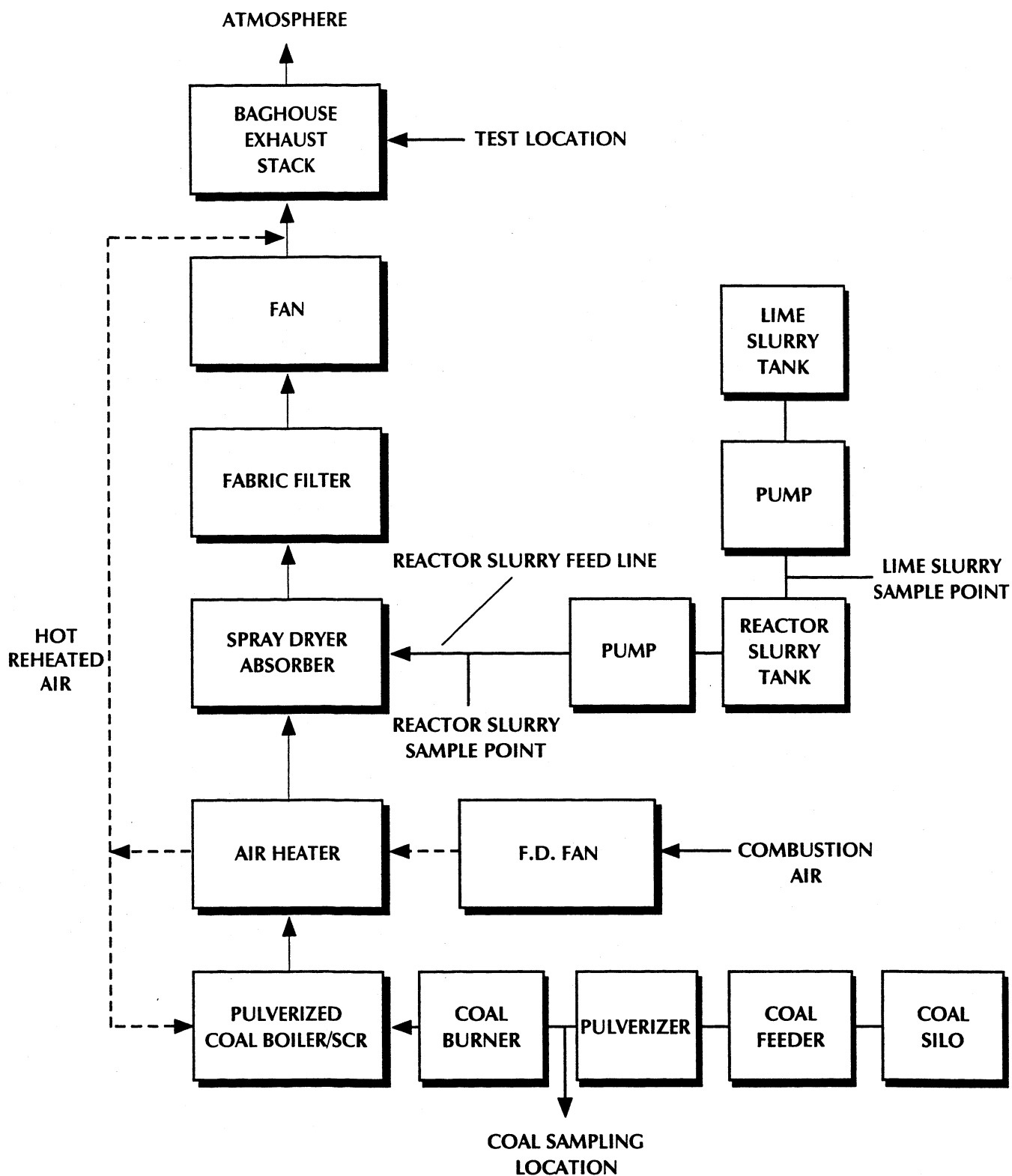


Figure 2-1. Process Air Flow Schematic

## **2.2 FLUE GAS SAMPLING LOCATIONS**

The sampling was conducted at the inlet to the spray dryer absorber and the baghouse exhaust stack.

### **2.2.1 Spray Dryer Absorber Inlet Location**

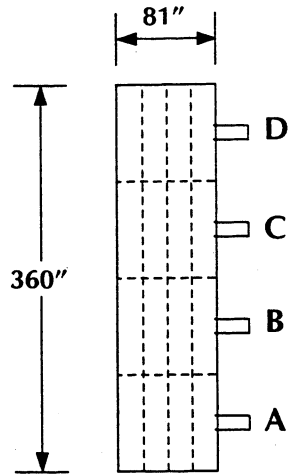
The spray dryer inlet sampling location is shown in Figure 2-2. The vertical rectangular duct is 81"x 360". The equivalent diameter for the SDA inlet duct is 132.2". The test ports are located approximately 288" (2.2 diameters) downstream from the nearest disturbance and approximately 360" (2.7 diameters) upstream from the nearest disturbance. The EPA Method 1 criteria were met and the maximum number of points (24) was sampled for each. Six (6) sampling points were traversed in each port. Table 2-1 presents the inlet traverse points. The spray dryer absorber inlet duct sampling location is fitted with four 4-inch ID ports that were used for testing.

**TABLE 2-1. SPRAY DRYER ABSORBER INLET TRAVERSE SAMPLING POINTS**

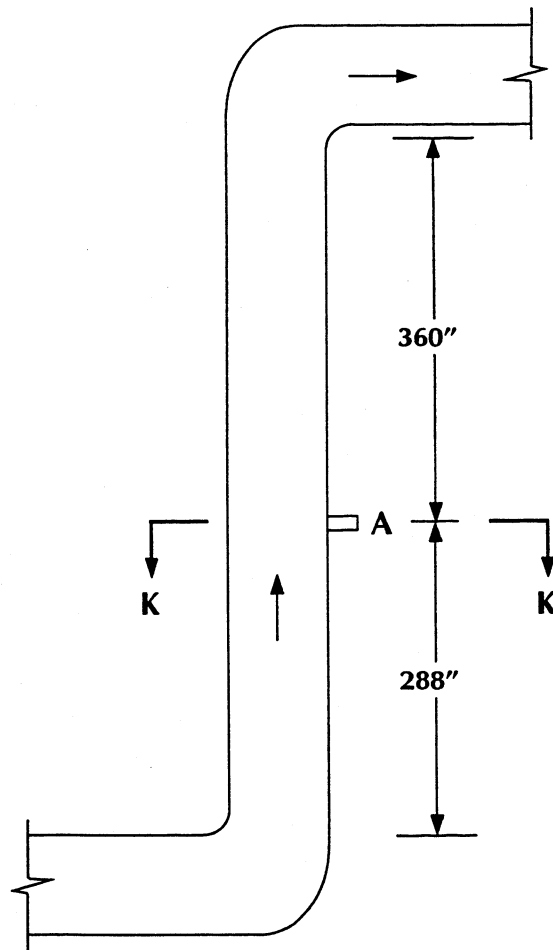
<b>Point</b>	<b>Distance from Wall, Inches</b>
1	6.75
2	20.25
3	33.75
4	47.25
5	60.75
6	74.25

### **2.2.2 PC Boiler Baghouse Exhaust Stack Location**

The PC Boiler and associated FGCS discharge to an exhaust stack approximately 400 feet tall. The sampling location is 214 feet above grade with a permanent test platform. At this elevation,



**SECTION K-K**



**Figure 2-2. SDA Inlet Test Location Schematic**

the test platform is serviced by an elevator. The exhaust stack has an inside diameter of 168 inches. Four 6-inch ports, located 90° apart on the same plane, are present on the stack. These ports are located 1,464 inches (8.7 stack diameters) downstream of the nearest disturbance and 2,106 inches (12.5 stack diameters) below the stack exhaust.

In accordance with EPA Method 1, TRC conducted a 12-point traverse (6-points on each diameter, 3 points per port) during each test run. Table 2-2 presents these traverse points. Figure 2-3 presents a schematic of the test location.

**TABLE 2-2. PC BOILER EXHAUST STACK TRAVERSE SAMPLING POINTS**

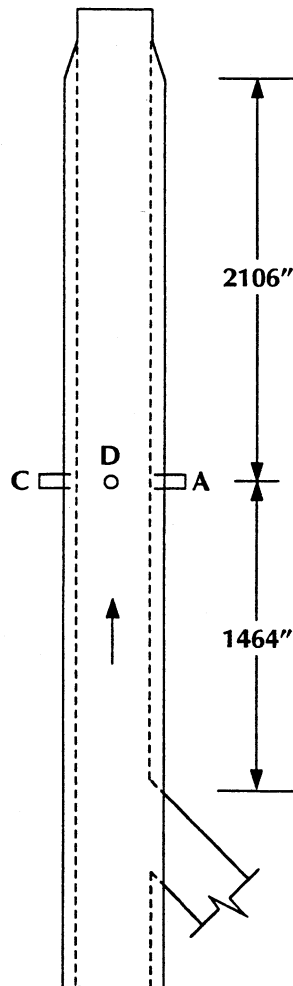
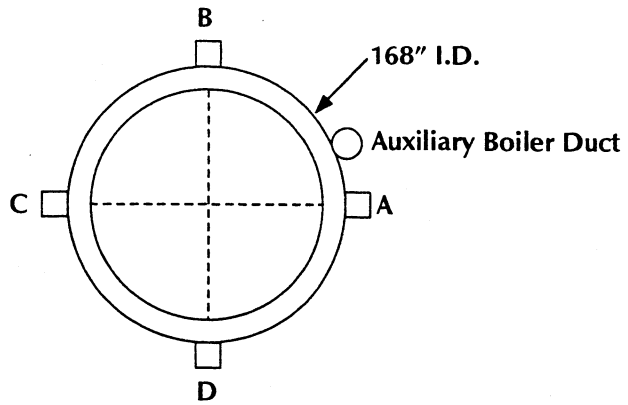
<b>Point</b>	<b>Percent of Stack Diameter</b>	<b>Distance From Wall (in.)</b>
1	4.4	7.39
2	14.6	24.53
3	29.6	49.73

## **2.3 PROCESS SAMPLING LOCATIONS**

During each sample run, as fired coal and as injected lime slurry were obtained at the following locations.

### **2.3.1 Coal Samples**

The unit coal feed system consists of 4 coal silos, 4 belt feeders, 2 pulverizers and 12 burner feed pipes. Each coal silo feeds a dedicated belt feeder. Two belt feeders feed one pulverizer. There are 3 exhaust pipes exiting each pulverizer with each exhaust pipe splitting into two burner pipes. Each burner pipe is fitted (as shown in Figure 2-4) with a coal sampling port. Coal was sampled from one burner pipe associated with each pulverizer.



Not to Scale

Figure 2-3. PC Boiler Sampling Location

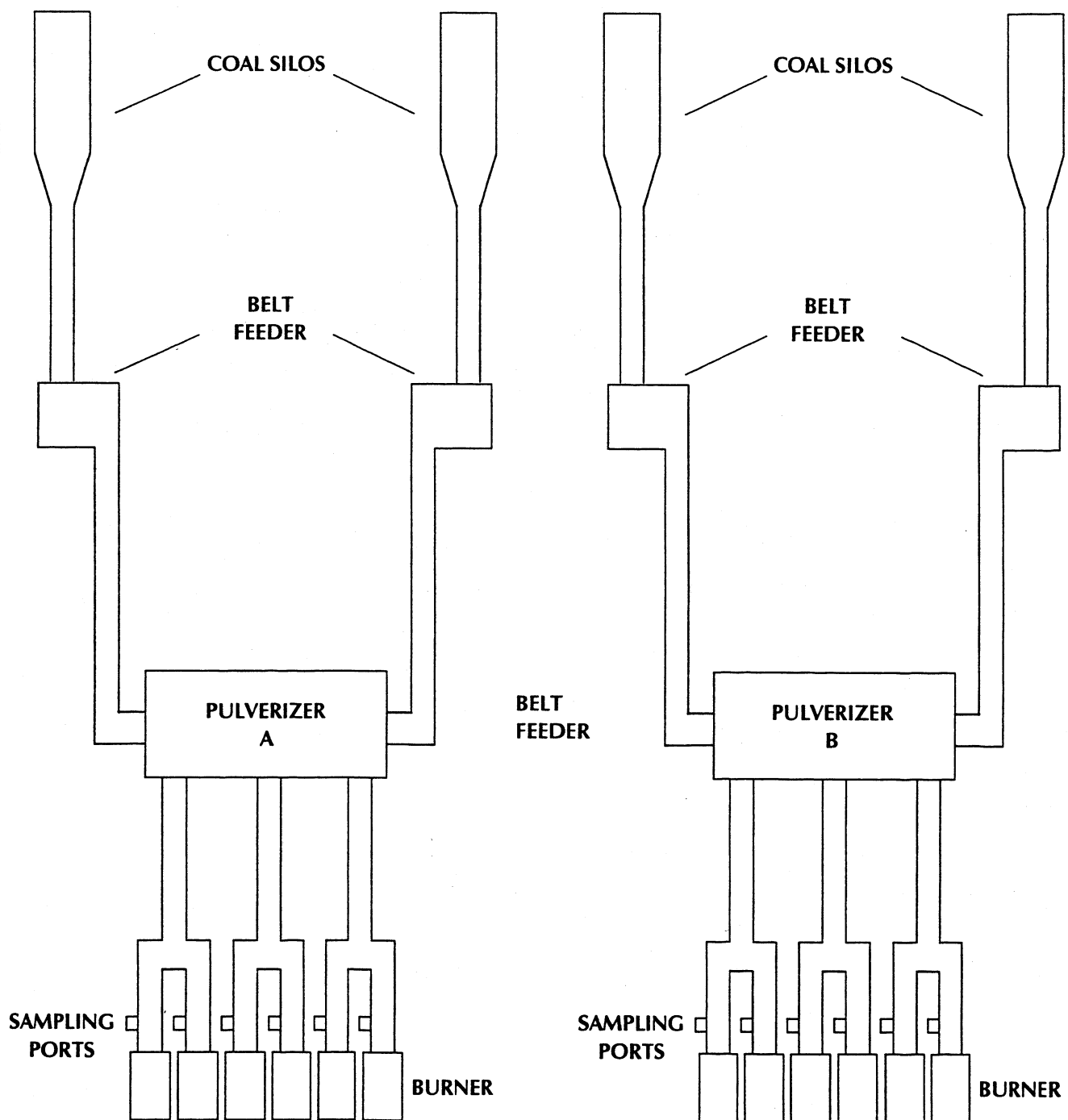


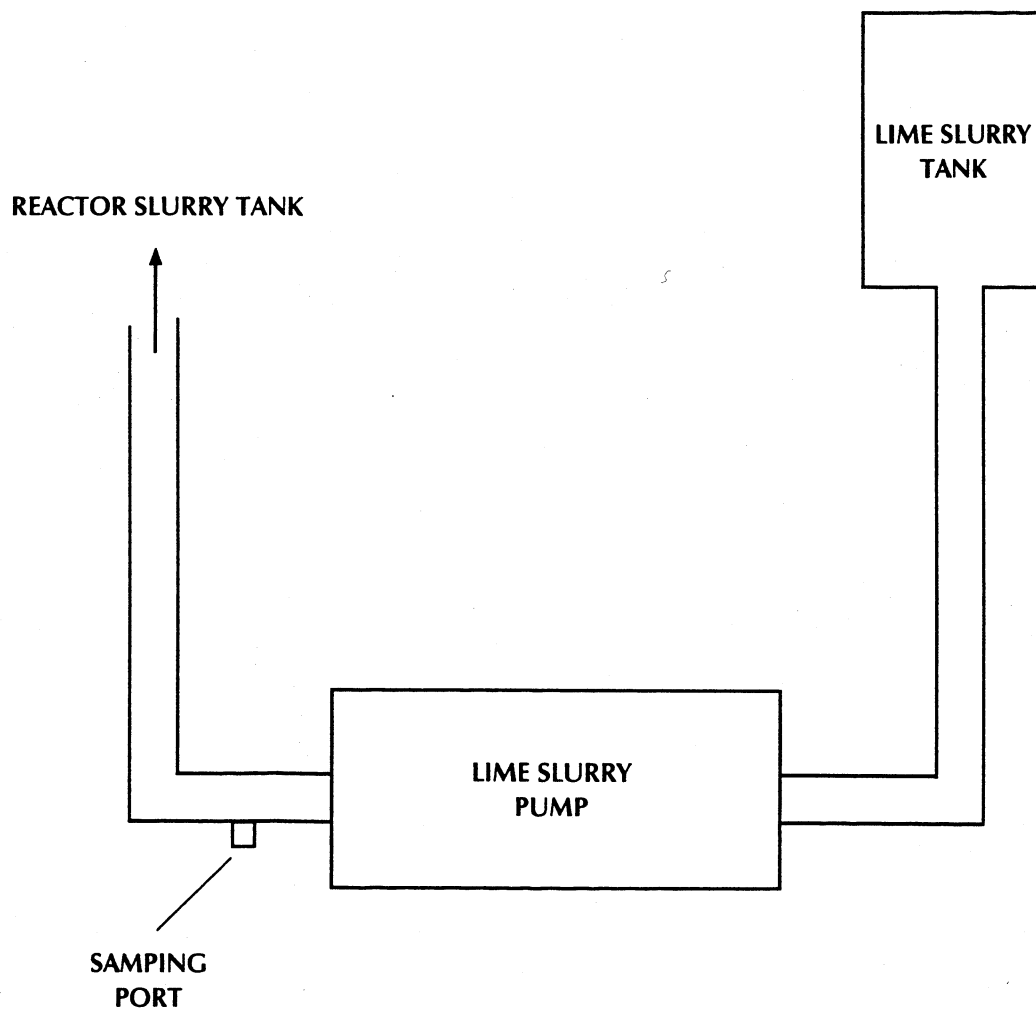
Figure 2-4. Coal Sampling Location

### **2.3.2 Lime Slurry Samples**

As injected lime slurry was obtained from the sampling port in the discharge lime slurry pump as shown in Figure 2-5. In addition to these samples, samples of the SDA reactor slurry were also collected. The reactor slurry was collected from the sampling port in the discharge of the reactor slurry pump which is shown in Figure 2-6.

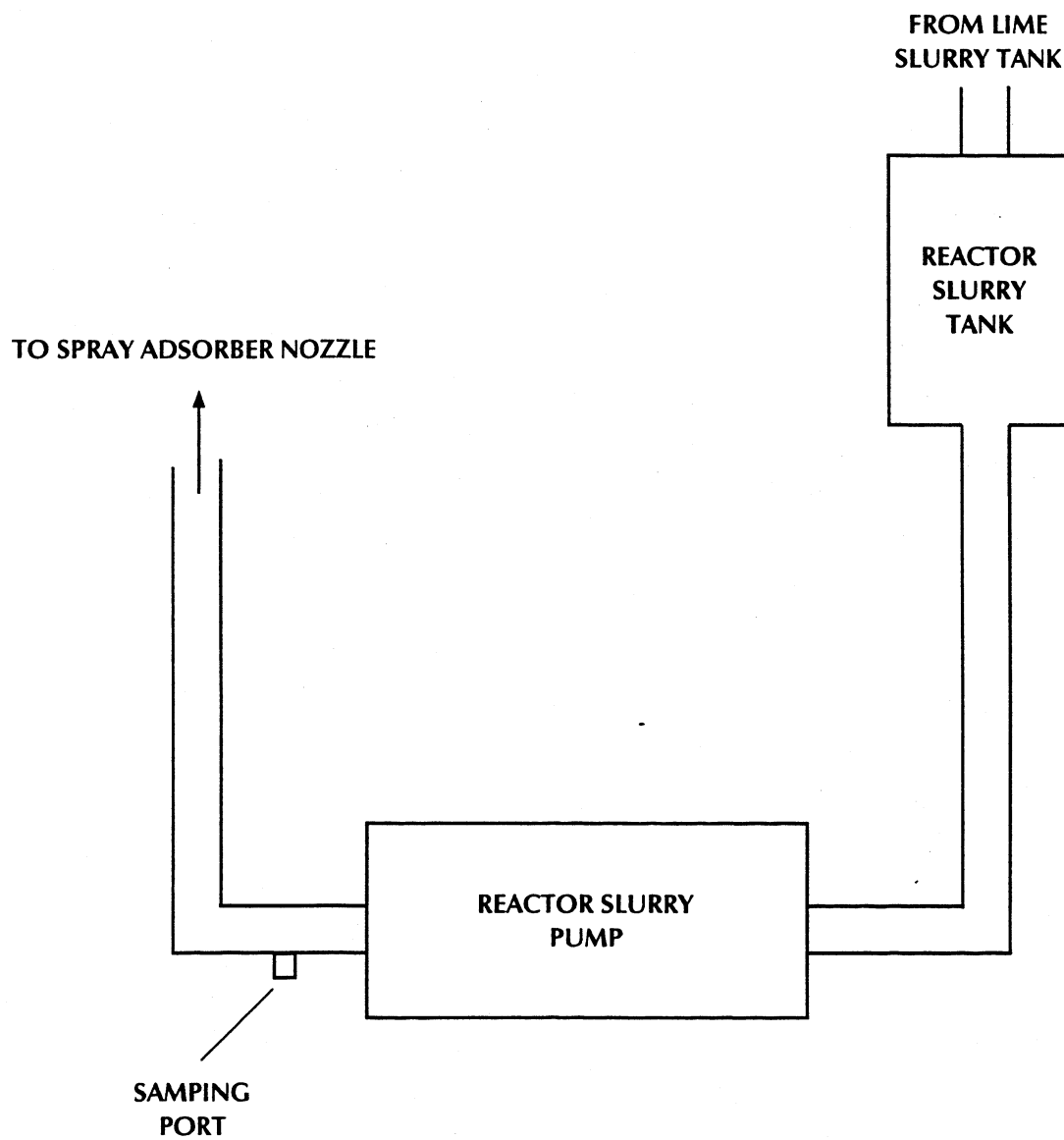
### **2.3.3 Aqueous Ammonia Sample**

As injected aqueous ammonia was obtained from the sampling port located on the storage tank as shown in Figure 2-7.

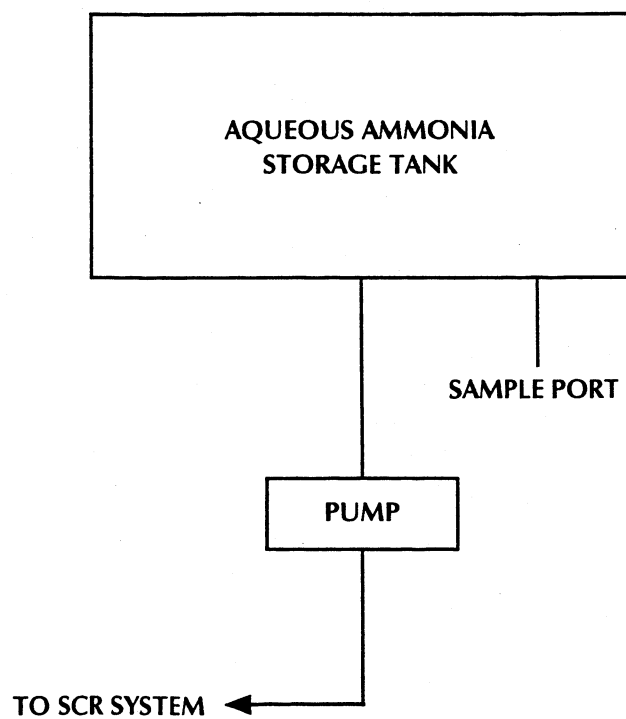


**Figure 2-5. Lime Slurry Sampling Location**





**Figure 2-6. Reactor Slurry Sampling Location**



**Figure 2-7. Aqueous Ammonia Solution Sample Location**

## SECTION 3.0

### SUMMARY AND DISCUSSION OF RESULTS

#### 3.1 OBJECTIVES AND TEST MATRIX

The objectives of the test program were as follows:

- measure the total inflow of mercury to the process;
- measure the concentration and emission rate of mercury on a speciated basis at the inlet and outlet of the facility pollution control equipment;
- determine the removal efficiency (RE) of the control equipment on a speciated and total basis;
- calculate an overall RE based upon the total inflow of mercury to the system; and
- utilize paired sampling trains at each location to evaluate method precision.

Table 3-1 presents the sampling and analytical matrix. Table 3-2 shows the measurements made at each test location. Precision evaluations made utilizing the paired sampling trains are presented in Section 5.4.1.4 of this report.

#### 3.2 FIELD TEST CHANGES AND PROBLEMS

##### 3.2.1 Field Test Changes

###### 3.2.1.1 *Inlet Sample Train Operation*

Due to the high negative static pressure encountered at the SDA inlet, the sample trains were started prior to insertion into the stack for each sample port traverse and were not shut down upon completion of a port traverse until after the nozzle had exited the sample port. This was necessary to avoid a back flush of the sample train and also to prevent the loss of any particulate matter during port changes. At the start of a sample port, the initial dry gas meter reading was taken as the nozzle of the sample train cleared the sample port sleeve and entered the gas stream. The final reading for a port was taken as the nozzle exited the gas stream and entered the sample

port sleeve. Taking the volume readings in this manner discounted the volume of air which passed through the train prior to entering or after exiting the stack.

#### **3.2.1.2      *Collection of Additional Process Samples***

Two additional process streams were sampled to determine total mercury inflow to the process. These included samples of the aqueous ammonia solution and the SDA “reactor slurry”. It was determined during the on-site pretest meeting that these were inflows to the system which were not taken into account in the SSTP and warranted sampling.

The reactor slurry consists of the slaked lime slurry to which is added fly ash from the baghouse. A composite sample was obtained for the reactor slurry in the following manner. Every 15 minutes a 500 mL grab sample was obtained from the sample valve downstream of the respective feed pumps. The lines were purged before obtaining each sample. The grab sample was placed in a 5 gallon container. At the completion of each run, the slurry in the container was mixed and a 1-liter sample collected for mercury analysis (SW846, 7471A).

A single 1-liter sample of the aqueous ammonia, utilized in the NO<sub>x</sub> SCR system, was obtained from a sampling valve located in the line exiting the storage tank. One sample was determined to be sufficient due to the fact that no ammonia was added to the storage tank prior to the completion of the sampling program. The sample was analyzed in accordance with SW846, 7470A.

#### **3.2.2   Problems**

This section documents problems that were encountered in the completion of the sampling program and the actions taken to overcome and/or correct the problems. None of the problems or actions taken are considered to have had a significant or negative impact upon the results reported.

### **3.2.2.1 Exhaust Stack Sampling Trains**

During the traverse of the first test port for Run 1, the dry gas meter utilized for sample train A developed a problem. It was determined after the completion of the first port that the meter was not indicating the full volume of air which had passed through the meter. This determination was based upon the fact that the total volume indicated by the A train meter was significantly lower than the volume recorded for the B sampling train. Since the trains were operated at nearly the same sampling rate, the volume recorded should have been similar. In addition, the volume recorded by the meter for the A train was lower than the volume which would be expected for the average sampling rate at which the train was operated.

The meter for the A train was replaced and the test continued. The volume collected during the traverse of the first port was calculated utilizing the average sampling rate ( $\Delta H$ ) recorded and the meter orifice calibration ( $\Delta H @$ ) for the meter in question. This volume was then added to the volume collected with the replacement meter during the remainder of the first test run. The equation utilized to calculate the sample volume is presented in Section 4.6 of this report. The problem with the meter was later determined to be a gear set screw which had loosened in the totalizer mechanism preventing proper advancement of the totalizer.

### **3.2.2.2 Inlet Sampling Trains**

Logistics at the SDA inlet were the cause of several minor problems with regard to sample train operation and port changes. The highly negative static pressure which exists at the location and its impact has been discussed previously in Section 3.1. The high static also contributed to the problems encountered. The problems encountered at the SDA inlet were as follows:

- During Run 1, the nozzle for inlet train A was dislodged and sucked into the stack while exiting port 2. The nozzle was lost. A leak check from the probe union was valid. A second nozzle was utilized to complete the run.

- During Run 1, during the final port change for train B, a glove was sucked into the port breaking the nozzle. A leak check from the probe union was valid. A second nozzle was utilized to complete the run.

In both cases it was determined that given the total mass of particulate collected by each sampling train, the potential loss of particulate matter resulting from breakage and/or loss of the nozzle was insignificant. Sample train isokinetics were calculated based upon the time period and sample volume collected for each nozzle. Section 4.6 presents the calculations utilized.

### **3.3 PRESENTATION OF RESULTS**

Presented in Tables 3-3 and 3-4 are the results required to fulfill the objectives identified for this test program. Table 3-5 presents the distribution of speciated mercury components measured at the SDA inlet and baghouse exhaust. The following sections discuss the results pertinent to each objective in greater detail. As discussed earlier, precision evaluations made utilizing the paired sampling trains are presented in Section 5.4.1.4 of this report.

#### **3.3.1 Total Mercury Inflow to the Process**

The total mercury inflow to the process was determined through sampling and analysis of the fuel and other materials utilized in the normal operation of the facility. These included the SDA slurry mixtures and the ammonia utilized for the SCR. Results are presented for each run and are based upon the concentration of mercury measured and the feed rate of each material into the process.

Table 3-3 presents the concentration of mercury measured in the process materials and the calculated feed rate (mg/hr) of mercury into the process. The sulfur and chlorine content of the fuel as well as the as fired HHV are also presented.

#### **3.3.2 Speciated and Total Mercury Emissions (Inlet and Outlet of Control Device)**

Concentrations and emission rates for mercury are presented in Table 3-3. Results are presented

on a speciated and total basis in terms of ug/dscm and mg/hr. Emission rates expressed in terms of lb/hrs and lbs/MMBtu are presented in Table 3-4. In the case where sample concentrations were below the Method Detection Limit (MDL) for a particular sample fraction, concentrations and emission rates were reported as “less than” (<) values. Although measurable quantities were reported by the laboratory for impingers 5 thru 7, the level of elemental mercury in the 4<sup>th</sup> impinger (HNO<sub>3</sub>/H<sub>2</sub>O<sub>2</sub>) was below the MDL for all sample trains. Therefore the overall results for elemental mercury were reported as less than values.

Paired trains were operated at each location for each of the three test runs. This resulted in a total of six samples collected at each location. Values presented in the summary tables (3-3, 3-3a) represent the average of the paired trains for a given sample location and run. Results for individual test runs are presented in Appendix A. Paired trains were operated in order to obtain a measure of precision for the speciated mercury method utilized for the test program. Section 5.4 of this report presents a comparison of the results obtained for the paired trains and a discussion of the measured precision.

An initial evaluation of the inlet and outlet data sets identified several data points which deviated from the others in their respective set of measurements. These data points were investigated to confirm the analytical results reported and identify any experimental conditions that may have contributed to the difference in reported values. Following confirmation of the analytical results and the determination that no difference in experimental conditions existed, the inlet and outlet data sets were each evaluated on a statistical basis. From these analyses, it was determined that several of the analytical results qualified as outlying values. As the footnote on Table 3-3 indicates, the results presented have been calculated excluding values from the data set that were determined to be outliers. Values reported for the following sample train fractions were discounted from the results reported in this section:

<u>Fraction</u>	<u>Sample Train ID</u>
Hg <sup>2+</sup>	Inlet Sample Trains - 1A and 2B
Hg <sup>0</sup>	Inlet Sample Trains - 1A, 3A; and Outlet Sample train - 3A

Appendix A contains a summary of the results incorporating all data points, inclusive of those determined to be outliers, as well as the calculations utilized in the statistical evaluation. Identification and treatment of outliers was accomplished in accordance with section B.10.4 of the Quality Assurance Program Plan (QAPP) prepared for this test program.

### **3.3.3 Emission Control Device Removal Efficiency**

The removal efficiency (RE) of the emission control equipment for speciated and total mercury was evaluated as part of the test program. RE calculations are based upon the emission rate measured for each of the speciated components at the baghouse exhaust stack and the SDA inlet sample locations.

As would be expected, the control device demonstrated a high efficiency for the particulate bound fraction, averaging 99.86% for the test program. RE for oxidized mercury ( $\text{Hg}^{2+}$ ) was determined to be 98.30% and that of elemental ( $\text{Hg}^0$ ) averaged 31.79%. The overall RE for mercury averaged 98.21%.

As discussed in Section 3.3.2, the results presented have been calculated excluding values from the data set that were determined to be outliers. Appendix A contains a summary of the results incorporating all data points as well as the calculations utilized in the statistical evaluation.

### **3.3.4 Overall Process Removal Efficiency**

The RE of the process for total mercury was evaluated as part of the test program. RE calculations are based upon the inflow of mercury to the system, based upon process monitoring, and the total mercury emission rate measured at the baghouse exhaust stack. The overall process RE for mercury averaged 98.53%.



**TABLE 3-1. PG&E GEN LOGAN GENERATING STATION TEST MATRIX**

Sampling Location	Sample/Type Pollutant	Sampling Method	Sampling Organization	No. of Trains at Location	No. of Runs Per Train	Sample Run Time (min)	Analytical Method	Analytical Laboratory
Inlet to Spray Dryer Absorber	Mercury speciated	Ontario Hydro (includes EPA M1, 2&4), M3B (Bag)	TRC	1	3	120	Ontario/Hydro & SW846, 7047A, CVAA	PhilLips Analytical Services
	O <sub>2</sub> /CO <sub>2</sub>		TRC	1	3	120	3B Orsat	TRC
Baghouse Exhaust Stack	Mercury Speciated	Ontario Hydro (includes EPA 1, 2 & 4) M3B (Bag)	TRC	1	3	120	Ontario/Hydro & SW846, 7470A, CVAA	Philips Analytical Services
	O <sub>2</sub> /CO <sub>2</sub>		TRC	1	3	120	3B Orsat	TRC
Coal Sampling	Sample	ASTM D2234-97a	PG&E Gen	-	3	120	ASTM 2013-86	Commercial Testing & Engineering
	Mercury Sulfur Chlorine GCV proximate moisture						D3684-94 D4234-92 D4208-88 D5865-98 D5142-90 D3302-97	
Lime Slurry	Mercury	ASTM D2334-97A	PG&E Gen	-	3	120	SW846, 7471A	Commercial Testing & Engineering
Reactor Slurry	Mercury	ASTM D2334-97A	PG&E Gen	-	3	120	SW846, 7471A	Commercial Testing & Engineering
Ammonia	Mercury	ASTM D2334-97A	PG&E Gen	-	1	-	SW846, 7470A	Commercial Testing & Engineering

**TABLE 3-2. MEASUREMENTS CONDUCTED AT EACH TEST LOCATION  
RUNS 1, 2 AND 3**

<b>Spray Dryer Absorber Inlet</b>	<b>Baghouse Exhaust Stack</b>	<b>Process</b>
EPA-EMC - Pre-003 Speciated Mercury - Ontario Hydro	EPA-EMC - Pre-003 Speciated Mercury - Ontario Hydro	Coal Feed Sample
O <sub>2</sub> /CO <sub>2</sub> (M3B)	O <sub>2</sub> /CO <sub>2</sub> (M3B)	Lime Slurry Sample
Sampling Location & Traverse Points (M-1)	Sampling Location & Traverse Points (M-1)	SDA Reactor Slurry Sample
Velocity (M-2)	Velocity (M-2)	Ammonia Sample
Moisture (M-4)	Moisture (M-4)	

**TABLE 3-3. SUMMARY TABLE OF EMISSIONS DATA AND REMOVAL EFFICIENCIES**

Method/Component	Units	SDA Inlet				Outlet Stack				Inlet/Outlet Removal Efficiency			
		1	2	3	Avg	1	2	3	Avg	1	2	3	Avg
<i>Ontario Hydro / Hg</i>													
Particle Bound	$\mu\text{g/dscm}$	12.30	12.39	11.90	12.20	0.0178	0.0162	0.0131	0.0157				
	mg/hr	9,467	9,373	9,090	9,310	14.70	13.50	10.66	12.95	99.84%	99.86%	99.88%	99.86%
Oxidized, Hg <sup>2+</sup>	$\mu\text{g/dscm}$	3.13*	1.87*	1.92	2.30	0.0319	0.0523	0.0156	0.0333				
	mg/hr	2,483*	1,382*	1,469	1,778	26.35	43.81	12.69	27.62	98.94%	96.83%	99.14%	98.30%
Elemental, Hg <sup>0</sup>	$\mu\text{g/dscm}$	<0.26*	<0.40	<0.29*	<0.31	<0.1801	<0.2022	<0.2022*	<0.1948				
	mg/hr	<203*	<301	<222*	<242	<148.58	<168.71	<167.20*	<161.50	26.86%	43.99%	24.52%	31.79%
Total, Hg	$\mu\text{g/dscm}$	<15.69	<14.65	<14.11	<14.81	<0.2298	<0.2707	<0.2309	<0.2438				
	mg/hr	<12,154	<11,056	<10,780	<11,330	<189.64	<226.02	<190.55	<202.07	98.44%	97.96%	98.23%	98.21%

Method/Component	Units	Process Samples				Overall Process Removal Efficiency			
		1	2	3	Avg	1	2	3	Avg
<i>Coal Samples</i>									
Hg	$\mu\text{g/g}$	0.19	0.18	0.17	0.18				
Hg Rate	mg/hr	14,482	13,735	12,955	13,724	98.69%	98.36%	98.53%	98.53%
Sulfur	Wt %	1.12	1.07	1.07	1.09				
Chlorine	%	0.15	0.15	0.15	0.15				
HHV	Btu/lb	13,758	13,767	13,757	13,761				
<i>Lime Slurry</i>									
	mg/L	<0.0002	<0.0002	<0.0002	<0.0002				
	mg/hr	<6	<7	<7	<7				
<i>Ammonia</i>									
	mg/L	<0.002	<0.002	<0.002	<0.002				
	mg/hr	<1	<1	<1	<1				
Total Hg Feed Rate	mg/hr	<14,489	<13,742	<12,962	<13,731				

\* Value represents data from one sample train. The corresponding value for the paired train was determined to be an outlying value with regard to all other data points for the given sample fraction and location.

TABLE 3-4. SUMMARY TABLE OF EMISSIONS DATA

Method/Component	Units	SDA Inlet				Outlet Stack			
		1	2	3	Avg	1	2	3	Avg
<i>Ontario Hydro / Hg</i> Particle Bound	lb/hr	2.06E-02	2.04E-02	1.98E-02	2.03E-02	2.89E-05	2.78E-05	2.49E-05	2.72E-05
	lb/MMBtu	9.13E-06	9.08E-06	8.88E-06	9.03E-06	1.46E-08	1.31E-08	1.05E-08	1.27E-08
Oxidized, Hg <sup>2+</sup>	lb/hr	5.40E-03*	3.01E-03*	3.20E-03	3.87E-03	5.73E-05	9.53E-05	2.76E-05	6.01E-05
	lb/MMBtu	2.33E-06*	1.37E-06*	1.43E-06	1.71E-06	2.61E-08	4.25E-08	1.25E-08	2.71E-08
Elemental, Hg <sup>0</sup>	lb/hr	<4.42E-04*	<6.55E-04	<4.82E-04*	<5.26E-04	<3.23E-04	<3.67E-04	<3.64E-04*	<3.51E-04
	lb/MMBtu	<1.91E-07*	<2.90E-07	<2.13E-07*	<2.32E-07	<1.47E-07	<1.64E-07	<1.63E-07*	<1.58E-07
Total, Hg	lb/hr	<2.64E-02	<2.41E-02	<2.35E-02	<2.47E-02	<4.10E-04	<4.90E-04	<4.16E-04	<4.39E-04
	lb/MMBtu	<1.17E-05	<1.07E-05	<1.05E-05	<1.10E-05	<1.88E-07	<2.19E-07	<1.86E-07	<1.98E-07

\* Value represents data from one sample train. The corresponding value for the paired train was determined to be an outlying value with regard to all other data points for the given sample fraction and location.

TABLE 3-5. DISTRIBUTION OF SPECIATED MERCURY EMISSIONS AND REMOVAL EFFICIENCIES

Method/Component	Units	SDA Inlet				Outlet Stack				Inlet/Outlet Removal Efficiency			
		1	2	3	Avg	1	2	3	Avg	1	2	3	Avg
<i>Ontario Hydro / Hg</i> Particle Bound	mg/hr	9,467 78%	9,373 85%	9,090 84%	9,310 82%	14.70 8%	13.50 6%	10.66 6%	12.95 6%	99.84%	99.86%	99.88%	99.86%
Oxidized, Hg <sup>2+</sup>	mg/hr	2,483* 20%*	1,382* 12%*	1,469 14%	1,778 16%	26.35 14%	43.81 19%	12.69 7%	27.62 13%	98.94%	96.83%	99.14%	98.30%
Elemental, Hg <sup>0</sup>	mg/hr	<203* <2%*	<301 <3%	<222* <2%*	<242 <2%	<148.58 <78%	<168.71 <75%	<167.20* <88%*	<161.50 <80%	26.86%	43.99%	24.52%	31.79%
Total, Hg	mg/hr	<12,154	<11,056	<10,780	<11,330	<189.64	<226.02	<190.55	<202.07	98.44%	97.96%	98.23%	98.21%

## SECTION 4.0

### SAMPLING AND ANALYTICAL PROCEDURES

#### 4.1 OVERVIEW

This section describes the procedures that TRC followed during the field sampling program. Throughout the program TRC followed 40 CFR, Part 60, Appendix A Methods and USEPA Preliminary Approved Sampling Protocols.

The remainder of this section is divided into several subsections: Field Program Description; Presampling Activities, Onsite Sampling Activities, Process Monitoring, Analytical Procedures, and Calculations.

The following test methods were utilized:

- EPA EMC Pre-003      Draft Standard Test Method for Elemental, Oxidized, Particle Bound Mercury Emissions in Flue Gas Generated From Coal-Fired Stationary Sources (Ontario Hydro Method) 7/7/99 revision.
- EPA Method 1      Sample Velocity Traverse for Stationary Sources
- EPA Method 2      Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S pitot tube)
- EPA Method 3B      Gas Analysis for the Determination of Emission Rate Correction Factor or Excess Air
- EPA Method 4      Determination of Moisture Content in Stack Gases
- ASTM D2234-97a      Standard Practice For the Collection of Representative Samples of Coal
- ASTM D-197      Standard Test Method for Sampling and Fineness Test of Pulverized Coal

## **4.2 PRESAMPLING ACTIVITIES**

Presampling activities included equipment calibration, precleaning of the sample train glassware, and other miscellaneous tasks. Each of these activities are described or referenced in the following subsections. Other presampling activities included team meetings, equipment packing, and finalization of all details leading up to the coordinated initiation of the sampling program.

### **4.2.1 Equipment Calibration**

See Section 5.0, Quality Assurance and Quality Control, of this Final Report.

### **4.2.2 Glassware Preparation**

The sample train glassware and sample containers required specialized precleaning to avoid contamination of the sample from the collection container or devices.

The Ontario Hydro sampling train glassware was precleaned with analconox soap and water wash. The glassware was rinsed with tap water, followed by three additional rinses with deionized water. The glassware was then soaked in a 10 percent nitric acid solution for 4 hours, rinsed three times with deionized water, and a final rinse with acetone. The glassware was then air dried and sealed with parafilm.

## **4.3 ONSITE SAMPLING ACTIVITIES**

Onsite sampling activities included equipment set up and conducting simultaneous testing of the SDA Inlet and Baghouse exhaust stack.

### **4.3.1 EPA Methods 1 and 2 for Velocity Measurements and Cyclonic Flow**

Velocity traverses were conducted at all sampling locations with an S-type pitot assembly in

accordance with 40 CFR Part 60, Appendix A, Method 1 "*Sample and Velocity Traverses for Stationary Sources*" and Method 2 "*Determination of Stack Gas Velocity and Volumetric Flow Rate (Type S Pitot Tube)*". An S-type pitot tube with an attached inclined manometer was used to measure the gas velocities. An attached Type-K thermocouple with a remote digital display was used to determine the flue gas temperature. During the test program, velocity measurements were conducted during each test run at each sampling location. The required number of velocity measurement points for each sampling location was determined following EPA Method 1.

Cyclonic flow checks were conducted at each sampling location prior to sampling in accordance with Section 2.4 of EPA Method 1. This procedure is referred to as the nulling technique. An S-type pitot tube connected to an inclined manometer was used in this method. The pitot tube was positioned at each traverse point so that the face openings of the pitot tube were perpendicular to the stack cross-sectional plane. This position is called the "0° reference". The velocity pressure ( $\Delta P$ ) measurement was noted. If the  $\Delta P$  reading was zero, the cyclonic angle was recorded as 0°. If the  $\Delta P$  reading was not zero, the pitot tube was rotated clockwise or counter clockwise until the  $\Delta P$  reading became zero. This angle was then measured with a leveled protractor and reported to the nearest degree. After this null technique was applied at each traverse point, the average of the cyclonic angles was calculated. If this average was less than 20°, the flow condition in the source was acceptable to test.

#### **4.3.2 EPA Method 4 for Moisture**

Moisture was determined for each test run according to EPA Reference Method 4, "*Determination of Moisture Content in Stack Gases*," as an integral part of the Ontario Hydro Method. The principle of this method is to remove the moisture from the sample stream and determine the moisture either volumetrically or gravimetrically.

Prior to the test program, a preliminary Method 4 was conducted at each sampling location to determine moisture and allow for the calculation of isokinetic sampling ratios. This sampling train used a glass lined probe with a thermocouple and S-type pitot tube attached to the probe for the



measurement of gas temperature and velocity. The sample gas passed through a series of four ice-cooled impingers kept below 68°F to enable condensation of entrained moisture. The first two impingers contained 100 mL of deionized water. The third impinger was empty and the fourth impinger contained a preweighed amount of silica gel. The impingers were followed by a dry gas meter, pump, and calibrated orifice meter. All impingers were weighed prior to the setup of the train.

Leak checks of the entire Method 4 sampling train were performed before and after each sampling run. All leak checks and leakage rates were documented on the relevant field test data sheet. The acceptance criterion for the Method 4 train was a leak rate of  $\leq 0.02$  cfm at the highest vacuum obtained during the run.

Following the completion of the preliminary test run, the Method 4 train was transported to a recovery area onsite. The sample recovery sequence was as follows:

- Removed the sampling train to the recovery area;
- Noted the condition of the train (i.e., impinger contents color, silica gel color, etc.); and
- The final weight of all impingers were obtained.

#### **4.3.3 Ontario Hydro Mercury Speciation Train (EPA EMC Pre-003)**

Speciated mercury (Hg) was determined at the SDA inlet and at the baghouse exhaust stack via EMC Pre-003 “*Draft Standard Test Method for Elemental, Oxidized, Particle Bound, and Total Mercury Emissions in the Flue Gas Generated From Coal Fired Stationary Sources (Ontario Hydro Method)*”. The description of the sampling and analytical methodology in this section is based on the draft method released July 7, 1999.

The sampling train (see Figure 4-1) consisted of a heated stainless steel, glass-lined probe with a glass button-hook nozzle. A thermocouple and S-type pitot tube were attached to the probe for

the measurement of gas temperature and velocity. The inlet sampling trains contained a cyclone and flask prior to the filter holder.

The sample gas passed through the probe assembly to a heated tared glass fiber filter, on a Teflon filter support, contained in a borosilicate filter holder. The probe and the gases exiting the filter holder were maintained at either the stack temperature  $\pm 21^{\circ}\text{F}$  or at  $248^{\circ}\text{F} \pm 25^{\circ}\text{F}$  whichever was greater. Downstream of the heated filter, the sample gas passed through a series of eight ice bath cooled impingers, kept below  $68^{\circ}\text{F}$  to enable condensation of entrained moisture and the gaseous mercury species. The first, second and third impingers each contained 100 mL of a 1N KCl solution. The fourth impinger contained 100 mL of a 5%  $\text{HNO}_3$ /10%  $\text{H}_2\text{O}_2$  solution. The fifth, sixth, and seventh impingers each contained 100 mL of a 4%  $\text{KMnO}_4$ /10%  $\text{H}_2\text{SO}_4$  solution. The eighth impinger contained 200 - 400 gms of silica gel. All filled impingers were weighed prior to placing the impingers in the train. The impingers were followed by a leak free pump, dry gas meter and calibrated orifice meter.

The first, second, fourth, sixth, and eighth impingers were of the Modified Greenburg design. The third, fifth and seventh impingers were standard Greenburg Smith impingers. No silicone grease was used in the train.

Sampling was isokinetic ( $\pm 10\%$ ) with a sample volume of between 35.31 and 88.25 dscf (1-2.5 dscm) collected. All stack and train operating parameters were recorded at each sampling point.

The sampling duration for each location was as follows:

- At the SDA inlet, the sampling duration was 6 minutes at each of the 24 traverse points for a total duration of 144 minutes.
- At the baghouse exhaust stack, the sample duration was 12 minutes with sample parameters recorded every 6 minutes. This results in a total run time of 144 minutes for the 12 point traverse.

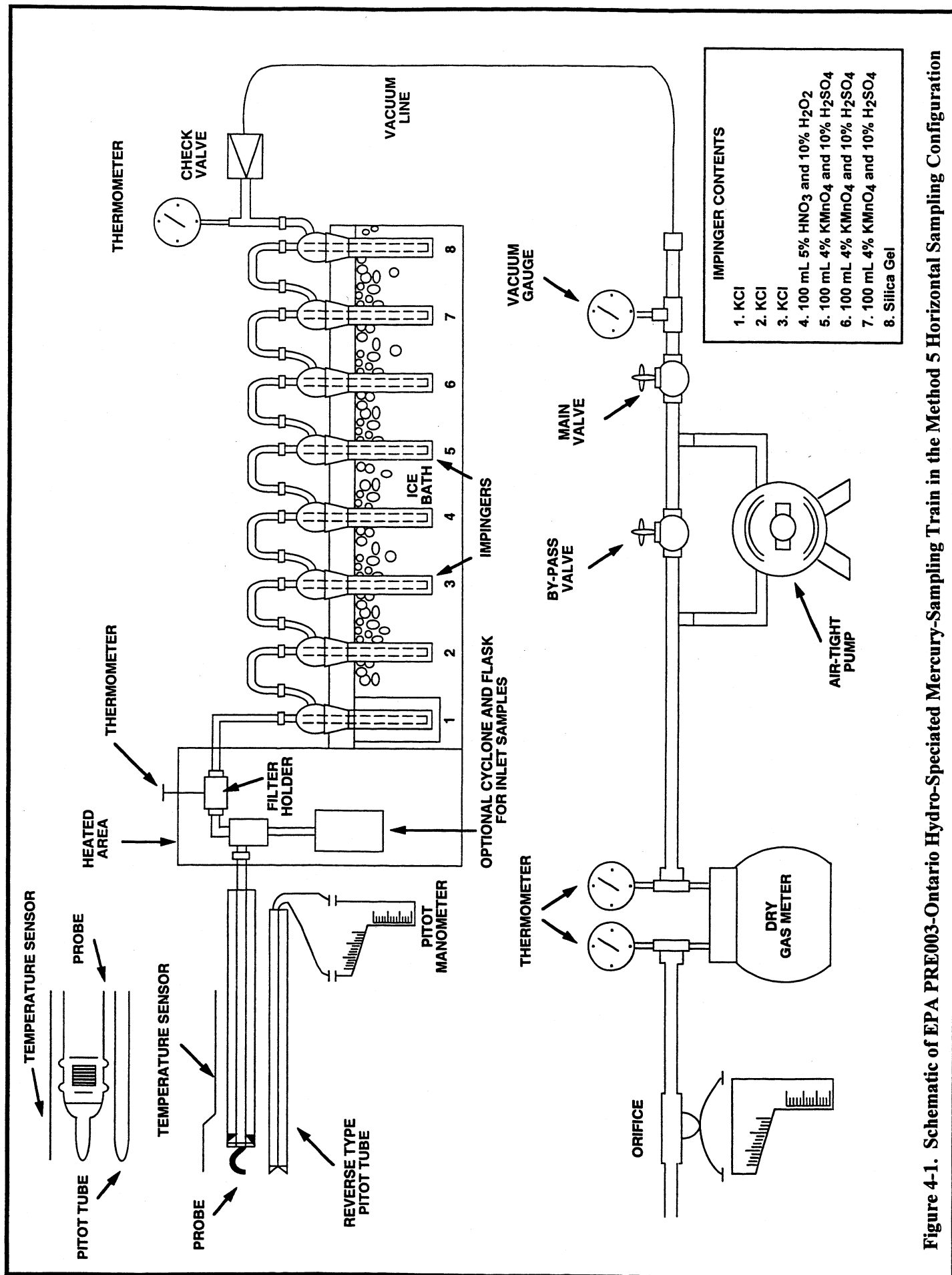


Figure 4-1. Schematic of EPA PRE003-Ontario Hydro-Speciated Mercury-Sampling Train in the Method 5 Horizontal Sampling Configuration

Leak checks of the entire Ontario Hydro sampling trains were performed before and after each sampling run. All leak checks and leakage rates were documented on the relevant field test data sheets. The acceptance criterion was a post run leak rate of  $\leq 0.02$  cfm at the highest vacuum obtained during the test run. The pre run leak check criterion was  $\leq 0.02$  cfm at 15 in Hg.

Following the completion of each test run, the Ontario Hydro train was transported to a recovery area onsite. The sample recovery sequence was as follows:

- All openings on the probe, inlet to cyclone/or filter holder and impingers were sealed with teflon tape. The sampling train was removed to the recovery area.
- The condition of the train was noted (i.e., filter, impinger contents color, silica gel color, etc.).
- Container No. 1 - Disassembled the filter housing and transferred the filter to its original glass petri dish. Sealed the petri dish with Teflon<sup>R</sup> tape and labeled it with the appropriate sample information. Any filter fibers adhering to the support gasket were transferred to the petri dish.
- Container 1B - The ash collected in the cyclone/flask from the inlet trains was transferred to a 250 mL amber wide mouth bottle. The bottle was sealed with teflon tape and labels.
- Container No. 2 - The front half of the train, nozzle, probe, and front-half filter housing, cyclone and flask were brush-rinsed with 100 mL of 0.1N nitric acid into an amber glass container with a Teflon<sup>R</sup>-lined cap. The container was sealed and labeled.
- Container No. 3 - The contents of the first three KCl impingers were weighed. The filter support, backhalf of the filter holder and connecting glassware were rinsed with 0.1 N HNO<sub>3</sub> into a glass amber container with a Teflon lined cap. The 5% KMnO<sub>4</sub> solution was added to each impinger until a purple color remained. The solutions were then poured into the container. The impingers and connecting glassware were rinsed with 10% HNO<sub>3</sub>. Although unlikely, if deposits remained on the impinger surfaces, they were removed by doing another 10% HNO<sub>3</sub> rinse that had a very small amount (several drops) of 10% hydroxylamine sulfate solution added to each of the KCl impingers. These rinses were added to Container 3. If the solution in Container 3 became clear, a small amount of the 5% KMnO<sub>4</sub> solution was added until a pink or slightly purple color was obtained. Checked again after 90 minutes to ensure that the purple color remained. Performed a final rinse of the impingers and connecting glassware with 0.1 N HNO<sub>3</sub> and added this rinse to Container 3. The container was sealed and labeled.

- Container No. 4 - The contents of the fourth impinger were weighed and transferred to a glass amber container with a Teflon<sup>®</sup>-lined cap. The impinger and U-tubes were rinsed twice with three 25 mL portions of 0.1N nitric acid into a sample container. The container was sealed and labeled.
- Container No. 5 (Impingers 5 through 7, H<sub>2</sub>SO<sub>4</sub>/KMnO<sub>4</sub> Impinger Contents and Rinses) - Dried the exterior surfaces of Impingers 5, 6, and 7. Then weighed and recorded the weight of each impinger (to the nearest 0.5 g). Poured all of the liquid from the three H<sub>2</sub>SO<sub>4</sub>-KMnO<sub>4</sub> impingers into a glass sample, Container 5. Rinsed the impingers and connecting glassware with a 0.1 N HNO<sub>3</sub>. If deposits remained on the impinger surfaces, after the two rinses, removed them by doing a third rinse with 0.1 N HNO<sub>3</sub> and several drops hydroxylamine sulfate. On a drop by drop basis added more hydroxylamine sulfate until the deposits were removed. Added these rinses to Container 5. If the solution in Container 5 became clear, added small amounts of H<sub>2</sub>SO<sub>4</sub>-KMnO<sub>4</sub> solution until a pink or slightly purple color was obtained. Performed a final 0.1 N HNO<sub>3</sub> rinse of the impingers and connecting glassware followed by a water rinse. The 0.1 N HNO<sub>3</sub> rinse was added to Container 5, and the water rinse was discarded. The container was sealed and labeled.
- The silica gel impinger was weighed to obtain a final weight.
- Solution Blanks (Containers 6 thru 10) - Solution blanks were taken each time new reagents were prepared.
- Container 6 - (0.1 N HNO<sub>3</sub> Blank) - Placed 50 mL of the 0.1 N HNO<sub>3</sub> solution used in the sample recovery process into a properly labeled container. Sealed the container.
- Container 7 (1 N KCl Blank) - Placed 50 mL of the 1 N KCl solution used as the impinger solution into a properly labeled container. Sealed the container.
- Container 8 (5% HNO<sub>3</sub> - 10% v/v H<sub>2</sub>O<sub>2</sub> Blank) - Placed 50 mL of the HNO<sub>3</sub>-H<sub>2</sub>O<sub>2</sub> solution used as the nitric acid impinger reagent into a properly labeled container. Sealed the container.
- Container 9 (H<sub>2</sub>SO<sub>4</sub> - KMnO<sub>4</sub> Blank) - Placed 50 mL of the H<sub>2</sub>SO<sub>4</sub> - KMnO<sub>4</sub> solution used as the impinger solution in the sample recovery process into a properly labeled container. Refer to Note 4 in Section 13.2.10.5 of this method.
- Container 10 (10% Hydroxylamine Sulfate Blank) - Placed 100 mL of hydroxylamine sulfate solution into a properly labeled sample container. Sealed the container.
- Container 11 (Sample Filter Blank) - Once during each field test, placed into a properly labeled petri dish three unused blank filters from the same lot as the sampling filters. Sealed the petri dish.

- All containers were checked to ensure proper sealing, proper labeling, and that all liquid levels were marked. All samples were logged onto a chain-of-custody record.

The Ontario Hydro train produced the following samples:

- Container No. 1 - Filter
- Container No. 1B - Ash (Inlet only)
- Container No. 2 - Front-Half 0.1N HNO<sub>3</sub> Rinse
- Container No. 3 - Impingers 1, 2 & 3 KCl Impinger Catch & Rinse
- Container No. 4 - Impinger 4 - 0.1N HNO<sub>3</sub> Impinger Catch & Rinse
- Container No. 5 - Impingers 5 - 7 - KMnO<sub>4</sub> Impinger Catch & Rinse

#### **4.3.4 EPA Method 3B for O<sub>2</sub> and CO<sub>2</sub>**

The O<sub>2</sub> and CO<sub>2</sub> concentrations in the integrated bag sample were analyzed onsite within four hours of the completion of the run with an Orsat analyzer as per EPA Method 3B, "*Gas Analysis for the Determination of Emission Rate Correction Factor or Excess Air*". Three or more passes were made until three results were within 0.2% (absolute) of each other.

#### **4.3.5 Process Sampling**

##### **4.3.5.1 Coal Sampling**

An integrated composite sample of as fired coal was obtained during each sampling run according to ASTM D-2234-97a and D197.

The subsamples were obtained from each of the two designated burner pipes with a cyclone sampler described in ASTM D197 every 15 minutes. The sampler probe was placed in the port of the burner pipe and drawn across the diameter and a quart mason jar was filled. At least six subsamples from each burner were obtained during a run. Each subsample was placed in a large

sample container which was capped between samples. The samples from both burners were combined into one composite sample and placed in a heavy wall plastic bag, sealed and labeled.

The composite sample was reduced at the analytical laboratory by riffing to an analytical and reserve sample at about 5 pounds each. The samples were placed in plastic heavy wall bags, sealed and labeled.

#### **4.3.5.2      *Lime Slurry Sampling***

An integrated slaked lime slurry composite sample was obtained from the sample port valve located in the discharge line of the lime slurry pump. A subsample was obtained every 15 minutes over the duration of the sample run. For each subsample, the sampling valve was purged for 15 seconds and then a 500 ml sample was obtained and placed in the composite bottle. After completing the run, the composite sample was mixed and a 1 liter analytical sample and reserve sample were taken. The bottles were sealed and labeled.

### **4.4      PROCESS MONITORING**

The facility operations data acquisition system (DAS) was utilized to record all operations data at one minute intervals. The data was averaged over the entire run period. Coal & limestone slurry feed were also totalized over the run.

Operations data collected by the facility is incorporated in Appendix G. The following process data was obtained.

Coal feeders	Feed rate K lbs/hr and totalizer reading for each feeder.
Limestone slurry	Feed rate K lbs/hr for each absorber
Electrical generation	KW
Main steam flow	K lbs/hr
Main steam temp.	°F

Main steam pressure	PSIG
Reheater steamer flow	K lbs/hr
Feed water flow	K lbs/hr
Furnace draft	in H <sub>2</sub> O
Wind box pressure	in H <sub>2</sub> O
Air heater temperature	in °F, out °F
Mills in operation	Total #
Burners in operation	Total #
Combustion air flow	K lbs/hr
Furnace O <sub>2</sub> /CO	%/ppm
Bag House temp.	°F
Baghouse Δp	in H <sub>2</sub> O
Limestone slurry - Nozzle pressure	PSIG
AQ Ammonia Inlet Flow	GPH

## 4.5 ANALYTICAL PROCEDURES

### 4.5.1 Mercury - Ontario Hydro Train Fractions

The Ontario Hydro train sample fractions samples were prepared according to Pre-003 and were analyzed for mercury by following the procedures in SW-846 Method 7470A. A schematic of the analytical process is shown in Figure 4-2.

The sampling train components were recovered and digested in the separate fractions. Materials collected in the sampling train were digested with acid solutions to dissolve inorganics and to remove organic constituents that may create analytical interferences. Acid digestion was performed using conventional or microwave digestion techniques.

All acid digested sample train fractions were analyzed for mercury by cold vapor atomic absorption spectroscopy (CVAAS) (SW 846 7470A).



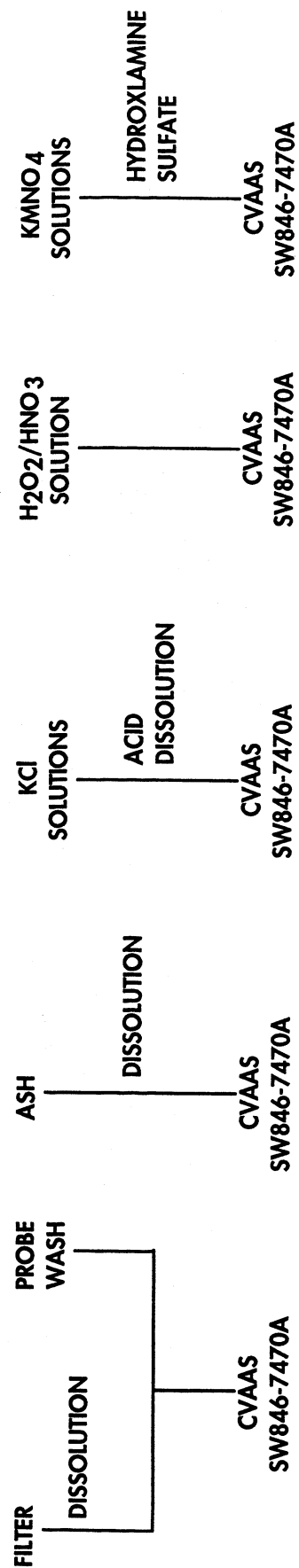


Figure 4-2. Analytical Scheme for EPA PRE003-Ontario Hydro Sampling Train Mercury Analysis

#### **4.5.2 Coal Analyses**

The samples were analyzed for sulfur, mercury, chlorine, total moisture, proximate analysis, and gross calorific value (GCV) by the methods delineated below.

##### Coal Sampling and Analysis

- ASTM D2234-97a Standard Practice for the Collection of Representative Samples of Coal.
- ASTM D2013-86 (1994) Standard method for Preparing Coal samples for Analysis.
- ASTM D3684-94 Standard Test Method for Total Mercury in Coal by the Oxygen Bomb Combustion /Atomic Absorption Method.
- ASTM D4208 -88 Standard Test Method for Chlorine in Coal by the Oxygen Bomb Combustion/Ion Selective Electrode Method.
- ASTM D3302-97a Standard Test Method for Total Moisture in Coal.
- ASTM D5142-90 Standard Test Method for Proximate Analysis of the Analysis Sample of Coal and Coke by Instrumental Procedures.
- ASTM D 4239-97 Standard Test Methods for Sulfur in the Analysis Sample of Coal and Coke Using High Temperature Tube Furnace Combustion Methods.
- ASTM D 5865-98 Standard Test Method for Gross Calorific Value Of Coal and Coke
- EPA SW846 7470A Mercury in Liquid Waste ( Manual Cold Vapor Technique).
- EPA SW846 7471A Mercury in Solid or Semi-Solid Waste (Manual Cold Vapor Technique).

#### **4.5.3 Lime Slurry**

The lime slurry was analyzed for mercury via SW846-7471A Mercury in Solid or Semi Solid Waste (Manual Cold Vapor Techniques).

## 4.6 CALCULATIONS

### 4.6.1 Flowrates and Isokinetics

The sequential calculations for the determination of gas velocity at stack conditions (afpm), gas volumetric flow rate at stack conditions (acfm), and gas volumetric flow rate at standard conditions (dscfm), and isokinetics found in 40 CFR 60 Appendix A Methods 1-5 are presented below.

#### *Calculations for Stack Volume and Isokinetic Ratio*

Time =	TT	T
Dry Gas Meter, ft <sup>3</sup> =	VM	
Pitot $\Delta P$ , in. H <sub>2</sub> O =	$\Delta P$	
Orifice $\Delta H$ , in. H <sub>2</sub> O =	PM	
Dry Gas Temp In, °F =	TMI	
Dry Gas Temp Out, °F =	TMO	
Stack Static Pressure, in. H <sub>2</sub> O =	PST	
Stack Temp, °F =	TS	

1. DN = Nozzle Diameter, inches
2. PB = Barometric Pressure, inches Hg
3. TT = Net Sampling Time, minutes
4. VM = VM final - VM initial = Sample Gas Volume, ft<sup>3</sup>
- 4A. VML = Use only if any final or intermediate leak check rate is over 0.02 cfm  
LI = Leak rate after any given sampling period, cfm  
TLI = Total time of sample period in which leak occurred, minutes  
VML = VM - [(L1 - 0.02) TLI + (L2 - 0.02) TL2 + (L3 - 0.02) TL3 + (L4 - 0.02) TL4] =

5. Average Dry Gas Temperature at meter, °F

$$TM = \frac{\text{Average TMI} + \text{Average TMO}}{2}$$

6. Average Orifice Pressure Drop, inches Hg

$$PM = \frac{\text{Average } \Delta H, \text{ in. H}_2\text{O}}{13.6}$$

7. Volume of dry gas sampled at standard conditions, dscf<sup>a</sup>

$$VMSTD = \frac{528 \times (Y) \times (VM) \times (PB + PM)}{29.92 \times (TM + 460)}$$

Y = dry gas meter calibration factor

8. Total Water Collected

$$VW = \text{gm H}_2\text{O silica gel} + \text{gm impinger H}_2\text{O}$$

Note: If ml H<sub>2</sub>O is measured - (ml × 0.9982 gm/ml = \_\_\_\_ gm)

9. Volume of water vapor at standard conditions, scf<sup>b</sup>

$$VW_{\text{gas}} = 0.04715 \times VW$$

10. Percent moisture in stack gas

$$\% M = \frac{100 \times VW_{\text{gas}}}{VMSTD + VW_{\text{gas}}}$$

- 10a. Percent moisture in stack gas - saturation (wet bulb/dry bulb method)

$$\% M = \frac{VP}{PS} \times 100$$

PS = Stack Pressure, absolute, inches Hg = PB ± Avg PST

PST = Stack static pressure

$$PST = \frac{\text{PST in. H}_2\text{O}}{13.6}$$

$$PS = PB \pm \text{Average PST}$$

$$TS_{\text{dry}} = \text{Stack Temperature, dry}$$

$$TS_{\text{wet}} = \text{Stack Temperature, wet}$$

Note: When  $TS_{\text{dry}} = TS_{\text{wet}}$ , the gas stream is saturated

SVP = water saturation vapor pressure at  $TS_{\text{wet}}$

$$VP = SVP - \left[ 0.00367 \times (PS) \times (TS_{dry} - TS_{wet}) \times \left( 1 + \left( \frac{TS_{wet} - 32}{1571} \right) \right) \right]$$

11. Mole Fraction of dry gas (dimensionless)

$$MD = \frac{100 - \%M}{100}$$

Note: The proper %M must be used in this calculation. The % vapor moisture can never be greater than the saturation value at given stack temperature. If 10 is greater than 10a, this is an indication of water droplets in the gas stream.

If 10 < 10a - use 10 %M in calculation

If 10 > 10a - use 10a %M in calculation

12. Molecular weight of dry stack gas

$$MWD = (\% CO_2 \times 0.44) + (\% O_2 \times 0.32) + [(\% CO + \% N_2) \times 0.28]$$

- 12a. % Excess Air

$$\%EA = \frac{[(\% O_2) - 0.5 \times (\% CO)] \times 100}{[(0.264) \times (\% N_2)] - (\% O_2) + 0.5 \times (\% CO)}$$

13. Molecular Weight of wet stack gas

$$MW = (MWD \times MD) + 18 \times (1 - MD)$$

14. AS = Stack Area, square inches

$$\text{Circular} = \left( \frac{\text{stack diameter}}{2} \right)^2 \pi$$

$$\text{Rectangular} = \text{Length} \times \text{Width}$$

15. PS = Stack Pressure, absolute, inches Hg = PB ± Avg PST

PST = Stack static pressure

$$PST = \frac{PST \text{ in. } H_2O}{13.6}$$

$$PS = PB \pm \text{Average PST}$$

16.  $TS_{avg}$  = Average Stack Temperature

$$17. \quad SDE_{avg} = \left( \sqrt{\Delta P} \right)_{avg} \times \sqrt{TS_{avg} + 460}$$

18. Stack gas velocity at stack conditions, afpm

$$VS = 5130^c \times C_p \times SDE_{avg} \times \left[ \frac{1}{PS \times MW} \right]^{1/2}$$

$C_p$  = pitot tube coefficient

19. Stack gas volumetric flow rate at stack conditions, acfm

$$Q_a = \frac{VS \times AS}{144}$$

20. Stack gas volumetric flow rate at standard conditions, dscfm<sup>e</sup>

$$Q_s = \frac{Q_a \times 528 \times MD \times PS}{(29.92) \times (TS_{avg} + 460)}$$

21. Percent Isokinetics

$$\%ISO = \frac{1039^f \times (TS_{avg} + 460) \times VMSTD}{VS \times TT \times PS \times MD \times (DN)^2}$$

<sup>a</sup> = Dry standard cubic feet at 68°F (528°R) and 29.92 in. Hg

<sup>b</sup> = Standard conditions at 68°F (528°R) and 29.92 in.Hg

<sup>c</sup> =  $5130 = \frac{ft}{85.5 \text{ sec}} \left[ \frac{(lb/lb-mole) \times (in. Hg)}{(^{\circ}R) \times (in. H_2O)} \right] \times 60 \text{ sec/min}$

<sup>d</sup> = Actual cubic feet per minute

<sup>e</sup> = Dry standard cubic feet per minute at 68°F (528°R) and 29.92 in.Hg

<sup>f</sup> =  $1039 = \frac{29.92 \text{ in. Hg}}{528^{\circ}R} \times \frac{144 \text{ in.}^2}{ft^2} \times \frac{4}{\pi} \times 100$

#### 4.6.1.1 Determination of VMSTD for Outlet Run 1A - Port 1

The following equation was utilized to calculate the VMSTD collected during the traverse of the first sample port for Outlet Run 1A:

$$VMSTD = \sqrt{\frac{\Delta H}{\Delta H@}} \times 0.75 \times t_{(min)} \times \left( \sqrt{\frac{528}{29.92} \times \frac{P_b + P_m}{T_{mout}}} \right)$$

This volume was then added to that measured during the traverse of the remaining sample ports for all subsequent calculations.

#### 4.6.1.2 *Determination of Time Weighted Nozzle Diameter (Use of Multiple Nozzles)*

The following equation was utilized to determine the time weighted average nozzle diameter:

$$DN_{TW} = \left( \frac{DN_1 \times TimeDN_1}{TotalTime} \right) + \left( \frac{DN_2 \times TimeDN_2}{TotalTime} \right)$$

The time weighted nozzle diameter was then utilized in the subsequent determination of the sample train isokenetic ratio.

#### 4.6.2 **Calculation for Particle Bound, Oxidized, Elemental and Total Mercury Concentrations**

The calculations for mercury species (i.e., as collected by the Ontario Hydro Sampling Train) are presented below. These are excerpted from Method Pre-003.

##### 4.6.2.1 *Particle-Bound Mercury*

##### 4.6.2.1.1 *Case 1: Amount of Ash on the Filter is Greater Than 0.5 g*

Calculate the concentration of mercury in ug/g in the ash sample ( $Hg_{ash}$ ) using Equation 8:

$$Hg_{ash}, \mu g / g = (IR)(DF) \quad [Eq. 8]$$

where:

IR = instrument reading,  $\mu g/L$

DF = dilution factor = (total digested volume, L)/(mass of ash digested, g)

Calculate the amount of mercury in the probe rinse ( $Hg_{pr}$ , Container 2) in  $\mu g$  using Equation 9:

$$Hg_{pr}, \mu g = (IR)(V_1) \quad [Eq. 9]$$

where:

IR = instrument reading,  $\mu g/L$

$V_1$  = total volume of probe rinse sample from which sample aliquot was taken, L.

Calculate the amount of mercury on the sample filter blank ( $Hg_{fb}$ ) in the same way using Equation 10:

$$Hg_{fb}, \mu g = (IR)(V_2) \quad [Eq. 10]$$

where:

IR = instrument reading,  $\mu g/L$

$V_2$  = total volume of sample filter blank digest, L.

The total amount of particle-bound mercury ( $Hg_{tp}$ ) then is determined using Equation 11:

$$Hg(\text{particle}), mg = (Hg_{ash})(W_{ash}) - Hg_{fb} + Hg_{pr} \quad [Eq. 11]$$

where:

$W_{ash}$  = the total ash weight on filter, g



The concentration of particle-bound mercury ( $\mu\text{g}/\text{dscm}$ ) in the gas stream is then determined using Equation 12:

$$\text{Hg}^{\text{tp}}, \mu\text{g} / \text{dscm} = \text{Hg}(\text{particle}) / V_{\text{m}(\text{std})} \quad [\text{Eq. 12}]$$

where:

$V_{\text{m}(\text{std})}$  = total volume of dry gas sampled at standard (normal) conditions, dscm

#### 4.6.2.1.2 Case 2: Amount of Ash on the Filter is Less than 0.5 g

The calculation is the same as in Case 1 except the entire sample (ash and filter) is digested; therefore, DF in Equation 8 is defined only by the total digested volume. Equations 9-11 remain the same.

#### 4.6.2.2 Oxidized Mercury

##### 4.6.2.2.1 KCl Solution (Impingers 1-3)

Calculate the concentration of mercury in  $\mu\text{g}/\text{L}$  in the KCl impinger solutions using Equation 13:

$$\text{Hg}_{\text{KCl}}, \mu\text{g} / \text{L} = (\text{IR})(\text{DF}) \quad [\text{Eq. 13}]$$

where:

IR = instrument reading,  $\mu\text{g}/\text{L}$

DF = dilution factor =  $V_{\text{D}} + V(\text{H}_2\text{SO}_4) + V(\text{HNO}_3) + V(\text{KMnO}_4) + V(\text{K}_2\text{S}_2\text{O}_8) + V(\text{NH}_4\text{OH})$   
 $V_{\text{D}}$

$V_{\text{D}}$  = total digested volume, 10 mL

$V(\text{H}_2\text{SO}_4)$  = volume of added concentrated  $\text{H}_2\text{SO}_4$ , 0.5 mL

$V(\text{HNO}_3)$  = volume of added concentrated  $\text{HNO}_3$ , 0.5 mL

$V(\text{KMnO}_4)$  = volume of added 5% w/v  $\text{KMnO}_4$ , 1.5 mL

$V(K_2S_2O_8)$  = volume of added 5% w/v  $K_2S_2O_8$ , 0.75 mL

$V(NH_2OH)$  = volume of added 10% w/v hydroxylamine sulfate, 1.0 mL

The amount of mercury in the KCl solution blank is calculated in the same way.

#### 4.6.2.2.2 *Total Oxidized Mercury (Hgo)*

Total Oxidized Mercury (Hgo) is defined by method as the mercury measured in the KCl sample minus the mercury measured in the KCl solution blanks as shown in Equation 14:

$$Hg_o, \mu g = (Hg_{KCl})(V_3) - (Hg_{Ob})(V_3) \quad [Eq. 14]$$

where:

$Hg_{KCl}$  = Mercury concentration measured in KCl aliquot,  $\mu g/L$

$V_3$  = Total volume of aqueous KCl from which sample aliquot was taken, L

$Hg_{Ob}$  = Mercury concentration measured in KCl solution blank aliquot,  $\mu g/L$

The concentration of  $Hg^{2+}$  ( $\mu g/dscm$ ) in the gas stream is then determined using Equation 15:

$$Hg^{2+}, \mu g / dscm = Hg_o / V_{m(std)} \quad [Eq. 15]$$

where:

$V_{m(std)}$  = Total volume of dry gas sampled at standard conditions, dscm

#### 4.6.2.3 *Elemental Mercury*

##### 4.6.2.3.1 *HNO<sub>3</sub>-H<sub>2</sub>O<sub>2</sub> Solution (Impinger 4)*

Calculate the concentration of mercury in  $\mu g/L$  in the  $HNO_3$ - $H_2O_2$  impinger solution using Equation 16:

$$\text{Hg}_{\text{H}_2\text{O}_2}, \mu\text{g} / \text{L} = (\text{IR})(\text{DF}) \quad [\text{Eq. 16}]$$

where:

IR = instrument reading,  $\mu\text{g}/\text{L}$

DF = dilution factor =  $\frac{V_D + V(\text{HCl}) + V(\text{KMnO}_4) + V(\text{K}_2\text{S}_2\text{O}_8) + V(\text{NH}_2\text{OH})}{V_D}$

$V_D$  = total digested volume, 5 mL

$V(\text{HCl})$  = volume of added concentration HCl, 0.25 mL

$V(\text{KMnO}_4)$  = volume of added saturated  $\text{KMnO}_4$ , mL (volume needed to turn sample to a purple color)

$V(\text{K}_2\text{S}_2\text{O}_8)$  = volume of added 5% w/v  $\text{K}_2\text{S}_2\text{O}_8$ , 0.75 mL (if used)

$V(\text{NH}_2\text{OH})$  = volume of added 10% w/v hydroxylamine sulfate, 1.0 mL

The amount of mercury in the  $\text{HNO}_3$ - $\text{H}_2\text{O}_2$  solution blank is calculated in the same way.

#### 4.6.2.3.2 $\text{H}_2\text{SO}_4$ - $\text{KMnO}_4$ Solution (Impingers 5-7)

Calculate the concentration of mercury in  $\mu\text{g}/\text{L}$  in the  $\text{H}_2\text{SO}_4$ - $\text{KMnO}_4$  impinger solutions using Equation 17:

$$\text{Mercury}, \mu\text{g} / \text{L} = \text{IR} \quad [\text{Eq. 17}]$$

where:

IR = instrument reading,  $\mu\text{g}/\text{L}$

Note - There is no dilution factor since no addition is made to the solution after the aliquot is taken for analysis.

The concentration of mercury in the  $\text{H}_2\text{SO}_4$ - $\text{KMnO}_4$  solution blank is calculated in the same way.

#### 4.6.2.3.3 Total Elemental Mercury ( $Hg_E$ )

Total Elemental Mercury ( $Hg_E$ ) is defined by method as the mercury measured in the  $H_2SO_4$ - $KMnO_4$  impingers plus the mercury in the  $HNO_3$ - $H_2O_2$  impingers minus the solution blanks as shown in Equation 18:

$$Hg_E, \mu g = (Hg_{H2O2})(V_4) - (Hg_{Eb1})(V_4) + (Hg_{KMnO4})(V_5) - (Hg_{Eb2})(V_5) \quad [Eq. 18]$$

where:

- $Hg_{H2O2}$  = Mercury concentration measured in  $HNO_3$ - $H_2O_2$  aliquot,  $\mu g/L$
- $V_4$  = Total volume of aqueous  $HNO_3$ - $H_2O_2$  from which sample aliquot was taken, L
- $Hg_{Eb1}$  = Mercury concentration measured in  $HNO_3$ - $H_2O_2$  solution blank aliquot,  $\mu g/L$
- $Hg_{KMnO4}$  = Mercury concentration measured in  $H_2SO_4$ - $KMnO_4$  aliquot  $\mu g/L$
- $V_5$  = Total volume of aqueous  $H_2SO_4$ - $KMnO_4$  from which sample aliquot was taken, L
- $Hg_{Eb2}$  = Mercury concentration measured in  $H_2SO_4$ - $KMnO_4$  solution blank aliquot,  $\mu g/L$

The concentration of  $Hg^0$  ( $\mu g/dscm$ ) in the gas stream is then determined using Equation 19:

$$Hg^0, \mu g/dscm = Hg_E / V_{m(std)} \quad [Eq. 19]$$

where:

- $V_{m(std)}$  = Total volume of dry gas sampled at standard conditions, dscm

#### 4.6.2.4 Total Mercury

Total mercury is defined by the method as the sum of the particulate bound mercury, oxidized mercury, and elemental mercury as shown in Equation 20:

$$Hg(\text{total}), \mu g/dscm = Hg^{tp} + Hg^{2+} + Hg^0 \quad [Eq. 20]$$

#### 4.6.3 Emission Rate of Mercury Species

The following equation is used for each species.

$$\text{lb/hr} = \frac{\mu\text{g/dscf mercury species} \times Q_s \text{ dscfm} \times 60 \text{ min/hour}}{453.59 \times 10^6 \mu\text{g/lb}}$$

$$\text{mg/hr} = \frac{\text{lb}}{\text{hour}} \times \frac{453,590 \text{ mg}}{\text{lb}}$$

#### 4.6.4 Mercury Removal Efficiency of Pollution Control System for each Species

$$RE = \frac{[(\text{total inlet mg/hr}) - (\text{outlet mg/hr})] \times 100}{\text{total inlet mg/hr}} = \%$$

#### 4.6.5 Coal and Limestone Analyses

The calculations for the concentration of pollutant in coal and limestone are given in each ASTM standard method. Pollutant concentrations were reported by the laboratory as follows:

Mercury	$\mu\text{g/g}$ , (ppm)
Chlorine	$\mu\text{g/g}$ , (ppm)
Sulfur	wt%, (lb/lb)
ash	wt%, (lb/lb)
moisture	wt%, (lb/lb)
gross heating value	Btu/lb

#### 4.6.6 Total Mercury Introduced into the Combustion Unit

Mercury From Process Streams:

$$\frac{mg}{hr} = (\mu g / g \text{ in feed}) \times \left( \frac{1000 lbs}{Klb} \right) \times \left( \frac{453,590 mg}{lb} \right) \times \left( \frac{1 mg}{10^3 \mu g} \right)$$

$$\frac{lb}{hr} = \frac{mg}{hr} \times \frac{1}{453,590 lb}$$

Total mercury to pollution control system:

$$mg/hr \text{ total} = mg/hr \text{ coal} + mg/hr \text{ reactor slurry} + mg/hr \text{ ammonia}$$

## SECTION 5.0

### QUALITY ASSURANCE

#### 5.1 OVERVIEW

Quality Assurance/Quality Control (QA/QC) protocols followed during this program were based on the procedures of the methods employed, as well as any additional measures outlined in the Quality Assurance Program Plan entitled; *"Quality Assurance Program Plan Mercury Emissions From Electric Utility Coal Fired Steam Generators Test Program US Generating Company"*, September 1999. Results of the QA/QC activities employed during this program are provided in this section.

As part of TRC's ongoing quality control for data reduction and reporting, all calculations are done using standardized EPA equations. TRC routinely reduces field data on a daily basis using a personal computer with software containing validated EPA equations. Isokinetics were determined at the end of each test day. Data such as those shown in the attached appendices were generated each day, with the exception of pollutant concentrations and emission rates, which were obtained after sample analyses were completed.

#### 5.2 FIELD QUALITY CONTROL SUMMARY

##### 5.2.1 Calibration Procedures

Calibration of the field sampling equipment was performed by TRC prior to the field sampling effort. Copies of the calibration sheets were submitted to the field team leader to take onsite and for inclusion in the project file. Calibrations were performed as described in the EPA publications *"Quality Assurance Handbook for Air Pollution Measurement systems; Volume III - Stationary Source Specific Methods,"* (EPA-600/4-77-027b) and EPA 40 CFR Part 60, Appendix A. Equipment that was calibrated included the sample metering system, nozzles, barometers,

thermocouples and pitot tubes. Pitot specific coefficients were determined for all pitots utilized during the test program by wind tunnel calibration in accordance with EPA Method 2 criteria. All calibrations were available for review during the test program. Copies of the equipment calibration forms can be found in Appendix B.

#### **5.2.2 Equipment Leak Checks**

Prior to sampling, each sampling train was leak checked according to the procedures outlined in EPA Reference Method 5. During the course of a test run, a leak check was conducted before and after every test or if replacement of a component became necessary. Final leak checks were performed to ensure that no leaks developed in the train during the course of the test run. All leakage rates were recorded on the isokinetic sampling data sheets presented in the appendices. Leak check results for all sampling trains met method acceptance criterion.

#### **5.2.3 Cyclonic Flow Check**

The absence of cyclonic flow at each sampling location was checked during preliminary traverses conducted prior to sampling, in accordance with Section 2.4 of EPA Method 1. Cyclonic flow was not found.

#### **5.2.4 Field Blanks**

Field blanks for both the inlet and outlet locations were taken during the setup day prior to the first test run. The field blanks were taken to each location, leak checked, and allowed to stay at the sampling location for the same time duration as a test run. At the completion of the time period, the blank trains were leak checked and brought down to the mobile laboratory for recovery. The glassware used for the field blanks was then utilized for Test Run 2 inlet and outlet samples.



### **5.3 SAMPLE HANDLING**

This section presents the sample handling, sample traceability, chain-of-custody (COC) procedures, sample transport and field documentation that TRC followed for the test program.

#### **5.3.1 Sample Traceability**

The purpose of sample traceability procedures was to document the identity of the sample and its handling from its first existence as a sample until analysis and data reduction were completed. Custody records traced a sample from its collection through all transfers of custody until it was transferred to the analytical laboratory. Internal laboratory records then documented the custody of the sample through its final disposition.

Sample integrity was maintained throughout all sampling and analysis programs. In accordance with SW-846, a sample was considered to be under a person's custody if the sample was:

- In that person's physical possession.
- In view of that person after acquiring possession.
- Secured by that person so that no one could tamper with the sample.
- Secured by that person in an area which was restricted to authorized personnel.

These criteria were used to define the meaning of "custody" and to ensure the integrity of the test program samples from collection to data reporting. Restricted access to the samples was an integral part of the COC procedure.

Samples were held within sight of the samplers or sample custodian, or were kept in sealed and secured containers at all times. Sealed containers were used to ship the samples to the laboratory.

### **5.3.2 Chain-of-Custody Documentation**

#### **5.3.2.1 *Labeling***

Sample identification labels were used by TRC to ensure that the required information was entered in the field. Sample labels were affixed to each appropriate process sample container for process samples at the time of collection. Exhaust gas sample labels were affixed to the appropriate container at the time of sample recovery. All samples collected during the test were labeled following the designated code system as stated in the Site Specific Test Plan (SSTP). Each sample label was preprinted prior to the test.

#### **5.3.2.2 *Field Logbook***

A permanently-bound field logbook was maintained by TRC's Field Team Leader. Information pertinent to the sampling was recorded in a sampling log. All entries were made in indelible ink and all corrections followed error correction protocol of one line through the error, initial of the person performing the correction and the date of the correction. Sampling personnel also recorded all information on the appropriate sampling forms.

#### **5.3.2.3 *Chain-of-Custody Forms***

To establish the documentation necessary to trace sample possession from the time of collection, a COC form was filled out (in four parts) and accompanied every sample or group of individually identified samples. Each person who had custody signed the COC form.

### **5.3.3 Sample Shipping**

Samples were packaged and shipped according to U.S. Department of Transportation, International Air Transportation Authority, and EPA regulations. Samples were delivered to the laboratory so that the requested analyses were performed within the specified allowable holding

time. Samples were accompanied by the COC form. The COC form listed the variables to be analyzed by the laboratory and the total number and type of samples shipped for analysis. Authorized laboratory personnel acknowledged receipt of shipment by signing and dating the COC form.

#### **5.4 LABORATORY QUALITY CONTROL SUMMARY**

As a routine QA/QC procedure, the laboratory analyzed blank and spike samples. The blank samples included laboratory reagents (method blanks), field blanks, and reagent blanks. Method blanks are used to measure any contaminants which may be introduced to the sample during sample handling in the laboratory. Field blanks are used to measure any contaminants which may be introduced to the samples from the sampling equipment and sampling technique.

Reagent blanks help measure any sample contamination which may have occurred in the reagents used to prepare and recover the sampling trains. The spike samples consisted of matrix spikes, matrix spike duplicates (MS/MSD) and blank spikes. The matrix and blank spikes were used to check the performance and the recovery efficiency of the various analytical methods used in this work.

The precision of analyses was measured by performing spikes and spike duplicates with the analytes of interest. The difference between duplicate analyses (MS/MSD) was used to estimate the precision of the analyses and the recovery of the spike samples was used to estimate the bias (accuracy) of the analysis.

The following subsections detail the Laboratory QC measures performed on the samples which were collected during this program.

##### **5.4.1 Mercury in Exhaust Gases**

Exhaust gases were sampled for mercury utilizing the Draft Ontario-Hydro Speciated Mercury

sample train. The analysis of the samples for mercury determination was accomplished using cold-vapor atomic absorption spectroscopy. Instrument calibration and calibration verification were performed in accordance with the above mentioned method.

#### **5.4.1.1      *Spike and Spike Duplicates***

The results of matrix spikes and matrix spike duplicates and a laboratory blank spike and blank spike duplicate prepared and analyzed along with the samples are presented in Table 5-1. The results presented in the table indicate that the analytical system was in control for the analysis of the samples.

#### **5.4.1.2      *Duplicate Analysis***

The results of the duplicate analysis of a prepared sample from both the inlet and outlet locations are presented in Table 5-2. The duplicate results indicate that precision of the instrument was within method criterion.

#### **5.4.1.3      *Blank Results***

Table 5-3 presents the results of the mercury analysis of the reagent and field blanks. As can be seen in Table 5-3, a significant level of mercury was detected in the field blank  $\text{KMnO}_4$  fraction and to a lesser degree, the KCl fraction. Review of the field blanks results with the field sample results have resulted in the following observations;

- The field  $\text{KMnO}_4$  sample results are lower than field blank results with the exception of Outlet Run 3a.
- The field sample trains show mercury concentrations substantially lower than the field blanks. The glassware used to set up the field blank trains was subsequently used to setup Inlet run 2 and Outlet Run 2. The lower results from these trains indicates that the glassware was not contaminated.

- The reagent blanks showed no contamination of the reagents used to make both the field blank and field sample trains.

Based on these observations, it has been concluded that there was no field wide mercury contamination and the results of the field blanks are not related to field conditions.

#### **5.4.1.4      *Paired Samples***

During this sampling program, paired samples were collected at both the inlet and outlet locations to the control device. Paired samples were obtained by collecting exhaust gas using two sampling trains at the same location at the same time. The results (reported in ug/dscm) of the paired samples are presented in Table 5-4. Although no precision criteria was stated in the QA plan for paired samples, TRC has used the limit of 50% relative percent difference (RPD) as an indicator that a loss in precision is occurring. In addition, the percent relative standard deviation (RSD) has been calculated for the entire data set to aid in the assessment of the precision of the entire data set.

The table shows that at least one component on each of the inlet train pairs has an RPD greater than 50%. Only one component in Outlet Run 3 was outside the 50% RPD limit. These high RPDs indicate a possible precision problem with this method. A more in-depth statistical analysis of the paired samples is presented in Appendix A.

#### **5.4.1.5      *Audit Sample Analysis***

As required by the Ontario-Hydro method, an audit sample was analyzed along with the samples. The audit sample was obtained from the National Institute for Standards and Technology (NIST). The audit sample was prepared and analyzed in duplicate with percent recoveries of 106% and 99% respectively. The recoveries of the audit sample analyses were well within acceptance limits of 90 - 110% recovery. The results of the audit sample analysis can be found in the analytical data package located in Appendix D.

#### **5.4.2 Analysis of the Process Feed Samples**

The process samples were analyzed for the parameters;

- Coal - mercury, sulfur, chlorine, and higher heating value,
- Lime Slurry - mercury,
- Ammonia - mercury.

The quality control data submitted with the analytical results indicate that the analytical process was within method specifications and the results should be considered valid.

**TABLE 5-1. SPIKE/SPIKE DUPLICATE RESULTS**

Sample ID(s): Inlet a Run 1									
Component	Spike Conc. (ug)	Sample Conc. (ug)	Measured Conc.		Percent Rec.				
			MS (ug)	MSD (ug)	MS	MSD	Limit	RPD	Limit
Fly Ash (mg/Kg)	0.97	0.97	1.9	N/S	96%		75-125%		20%
Front Half	9.7	9.5	19	19	98%	98%	75-125%	0.00%	20%
KCl	17.5	16	33	33	97%	97%	75-125%	0.00%	20%
KMnO4	3.45	3.7	7.0	7.1	96%	99%	75-125%	1.42%	20%
HNO3/H2O2	2.55	0.34	2.9	2.9	100%	100%	75-125%	0.00%	20%

Sample ID(s): Outlet a Run 2									
Component	Spike Conc. (ug)	Sample Conc. (ug)	Measured Conc.		Percent Rec.				
			MS (ug)	MSD (ug)	MS	MSD	Limit	RPD	Limit
Fly Ash (mg/Kg)									
Front Half	0.1	0.047	0.15	0.15	100%	100%	75-125%	0.00%	20%
KCl	0.46	0.081	0.56	0.55	104%	102%	75-125%	1.80%	20%
KMnO4	0.39	0.31	0.68	0.68	95%	95%	75-125%	0.00%	20%
HNO3/H2O2	2.6	0	2.6	2.7	100%	100%	75-125%	0.38%	20%

Sample ID(s): Blank Spike									
Component	Spike Conc. (ug)	Sample Conc. (ug)	Measured Conc.		Percent Rec.				
			MS (ug)	MSD (ug)	MS	MSD	Limit	RPD	Limit
Fly Ash (mg/Kg)									
Front Half	0.1	0	0.10	0.10	100%	99%	75-125%	1.01%	20%
KCl	0.46	0	0.45	0.47	100%	102%	75-125%	2.39%	20%
KMnO4	0.22	0	0.22	0.22	100%	100%	75-125%	0.00%	20%
HNO3/H2O2	0.1	0	0.10	0.10	100%	100%	75-125%	0.00%	20%

**TABLE 5-2. LABORATORY DUPLICATE RESULTS**

Field Sample ID.:	Inlet a Run 1	Reporting Limit	Inlet a Run 1	Reporting Limit		RPD
Component	(ug)	(ug)	(ug)	(ug)	RPD	Limit
Fly Ash (mg/Kg)	0.97	0.04	0.98	0.04	1.03%	25%
Front Half	9.5	0.01	9.4	0.01	1.06%	25%
KCl	16	0.03	16	0.03	0.00%	25%
KMnO4	3.7	0.03	3.7	0.03	0.0%	25%
HNO3/H2O2	0.34	0.01	0.3	0.01	12.50%	25%

Field Sample ID.:	Outlet a Run 2	Reporting Limit	Outlet a Run 2	Reporting Limit		RPD
Component	(ug)	(ug)	(ug)	(ug)	RPD	Limit
Fly Ash (mg/Kg)						
Front Half	0.047	0.01	0.047	0.01	0.00%	25%
KCl	0.081	0.03	0.081	0.03	0.00%	25%
KMnO4	0.31	0.03	0.31	0.03	0.0%	25%
HNO3/H2O2	0	0.01	0	0.01	NC	25%



**TABLE 5-3. BLANK RESULTS**

Field Sample ID.: Component	Reagent Blank (ug)	Inlet Field Blank (ug)	Outlet Field Blank (ug)
Front Half	<0.01	<0.01	<0.01
KCl	<0.030	0.083	0.21
KMnO4	0.04	1.8	2
HNO3/H2O2	<0.25	<0.25	<0.25

TABLE 5-4. PAIRED SAMPLE TRAIN RESULTS

Component	Run							
	Ia Run 1 ug/dscm	Ib Run 1 ug/dscm	RPD	Ia Run 2 ug/dscm	Ib Run 2 ug/dscm	RPD	Ia Run 3 ug/dscm	Ib Run 3 ug/dscm
Front Half (part.)	12.2	12.4	2	14.2	10.6	29	11.4	12.4
KCl	6.6	3.13	71	1.87	5.28	95	1.58	2.26
HNO <sub>3</sub> /KMnO <sub>4</sub>	1.66	0.256	147	0.374	0.418	11	1.05	0.29
Total Hg	20.4	15.8	26	16.4	16.3	1	14.0	14.9
								7
								14%
Component	Run							
	Oa Run 1 ug/dscm	Ob Run 1 ug/dscm	RPD	Oa Run 2 ug/dscm	Ob Run 2 ug/dscm	RPD	Oa Run 3 ug/dscm	Ob Run 3 ug/dscm
Front Half (part.)	0.019	0.017	9	0.019	0.014	33	0.014	0.012
KCl	0.024	0.040	48	0.033	0.071	73	0.016	0.015
HNO <sub>3</sub> /KMnO <sub>4</sub>	0.170	0.190	11	0.230	0.175	27	1.40	0.202
Total Hg	0.213	0.246	14	0.282	0.260	8	1.43	0.229
								145
								191%

Ia = Inlet train a Ib = Inlet train b Oa = Outlet train a Ob = Outlet train b  
 TRC advisory limit = 50% RPD.

